EDGEWOOD

CHEMICAL BIOLOGICAL CENTER

U.S. ARMY SOLDIER AND BIOLOGICAL CHEMICAL COMMAND

ECBC-TR-360

QUANTITATIVE INFRARED REFERENCE LIBRARY VOLUME II

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Christopher C. Keiser

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December 2003

Aberdeen Proving Ground, MD 21010-5424

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14. ABSTRACT
Recent technology developments in Fourier Transform Infrared (FTIR) spectroscopy (such as stand-off infrared (IR) detection of chemical agents and related compounds for monitoring applications) has created a need for condensed and vapor phase FTIR databases, which use current technology. Old IR databases are obsolete as they were collected on first generation instruments with poor signal-to-noise characteristics arising from a variety of instrumental variations (such as noisy A/D converters, wavelength calibration problems, etc.) relative to the capabilities of present day FTIR instrumentation. In addition, commercial IR spectra complications do not adequately address militarily significant or directly related compounds.

The objective of this effort is to create, validate, and maintain a vapor phase FTIR database, with a focus on compounds of military significance or potential terrorist threat, using instrumentation that reflects current analytical capabilities.

15. SUBJECT TERMS
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Chemical warfare agents
Infrared spectroscopy
Vapor phase spectra
Infrared spectra
FTIR

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PREFACE

The work described in this report was authorized under Project No. 2VJRZZ. The work was started in June 2002 and completed in December 2002.

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QUANTITATIVE INFRARED REFERENCE LIBRARY
VOLUME II

1. INTRODUCTION

Recent technology developments in Fourier Transform Infrared (FTIR) spectroscopy, such as stand-off infrared detection of chemical agents and related compounds for monitoring applications, has created a need for condensed and vapor phase FTIR databases which utilize current technology. Old infrared databases are obsolete as they were collected on first generation instruments with poor signal-to-noise characteristics arising from a variety of instrumental variations (such as noisy A/D converters, wavelength calibration problems, etc.) relative to the capabilities of present day FTIR instrumentation. In addition, commercial infrared spectra compilations do not adequately address militarily significant, or directly related, compounds.

2. OBJECTIVE

The objective of this effort is to create, validate, and maintain a vapor phase FTIR database, with a focus on compounds of military significance or potential terrorist threat, using instrumentation that reflects current analytical capabilities.

3. DESCRIPTION

A series of quantitative concentrations of the analytes were established with a Kin-Tek model 491-M gas/vapor generation system. The Kin-Tek generator uses chemical-filled ¼" o.d. polytetraflouroethylene permeation tubes contained in a glass holder to establish continuous streams of chemical compounds. The glass permeation tube holder sits in a heated oven block, regulated by a digital temperature controller. Balance gas, regulated by digital mass flow controllers, is supplied to the vapor stream at two points: the permeation tube holder and downstream of the permeation tubes to provide additional diluent. The output concentration of the device can be varied by either changing the temperature of the oven block or the flow rate of the diluent gas. For the series of experiments in this paper, dry nitrogen, supplied by boiloff from a 230-L liquid nitrogen Dewar, was used.

The rate of mass flow from permeation tubes can be determined gravimetrically, by weighing the tubes before and after an operation. Because of the low volatilities of the compounds in these tests, this would have required potentially several weeks for each agent in order to accurately measure the mass loss. Furthermore, chemical agents tend to be chemically "sticky" and can adsorb to tubing walls and fittings. For this reason, the mass rate was determined with a secondary method. Using a mass flow controller, a measured volume of the effluent was drawn onto a sorbent tube filled with Tenax-TA for later desorption and analysis by gas chromatography. Analysis of the tubes was performed on an Agilent 5890A gas

chromatograph (GC) equipped with a Dynatherm thermal desorption apparatus and a flame ionization detector. The instrument was calibrated prior to each operation with external standards, prepared by serial dilution from the neat material in hexane, which were injected onto a sorbent tube and then desorbed into the GC. Six tubes were collected for each operation (agent). A statistical analysis of the data from the sorbent tubes showed typical RSD's of better than 5%.

Transfer lines between the generator and the FTIR gas cell were plumbed with ¼" o.d. Silicosteel® from Restek Corporation. This material minimizes adsorption by a wide variety of chemical compounds, enabling a more rapid stabilization of the concentration of the analyte in the gas cell.

Reference spectra of the effluent from the generator were acquired with a Protegé model Fourier transform infrared (FTIR) spectrometer from Thermo-Nicolet. The spectrometer is equipped with a mercury-cadmium-telluride (MCT) detector operating at cryogenic temperatures (achieved by filling the detector with liquid nitrogen prior to operation). The spectrometer is also outfitted with a 10-meter fixed pathlength gas cell from Thermo-Spectratech. Spectral conditions were 0.5 cm^{-1} resolution, 3-term Blackman-Harris apodization, and co-adding of 64 scans to achieve the final spectra. Prior to any change in concentration, a fresh background (single beam) spectrum of the dry nitrogen was collected. As a quality check, a statistical analysis of the data was made, using peak heights to fit several representative bands at the vapor concentrations analyzed to a least squares fit, in order to determine compliance with Beer's Law.

APPENDIX

EDGEWOOD CHEMICAL BIOLOGICAL CENTER
QIRL DATABASE

Edgewood Chemical Biological Center
QIRL Database

QIRL Filename	Compound Name	CAS	Molecular Formula	CL (ppm-m)
QIRL0210	Pinacolone	75-97-8	(CH3)3CCOCH3	488
QIRL0220	Pinacolyl Alcohol	464-07-3	(CH3)3CCHOHCH3	170
QIRL0230	Diethyl Ethylphosphonate	78-38-6	(CH3CH2O)2P(O)CH2CH3	37
QIRL0240	N-Methyldiethanolamine	105-59-9	(HOCH2)2NCH3	10
QIRL0250	Cyanogen Chloride (CK)	506-77-4	CClN	343
QIRL0260	2-Furaldehyde	98-01-1	C5H4O2	74
QIRL0270	Trichloronitromethane (PS)	76-06-02	CCl3NO2	177
QIRL0280	2-(Diisopropylamino) ethanethiol	5842-07-9	C6H14NCH2CH2SH	20
QIRL0290	2-(Diethyamino) ethanethiol	100-38-9	C4H10NCH2CH2SH	72
QIRL0300	2-(Diethyamino) ethanol	100-37-8	C4H10NCH2CH2OH	173
QIRL0310	Benzyl alcohol	100-51-6	C6H5CH2OH	84
QIRL0320	Diisopropyl fluorophosphate	55-91-4	(C3H7O)2P(O)F	104
QIRL0330	Diethyl pimelate	2050-20-6	C2H5O2C(CH2)6O2C2H5	10
QIRL0340	Allyl isothiocyanate	57-06-7	C2CHCH2CNS	128
QIRL0350	Benzyl bromide	100-39-0	C6H5CH2Br	119
QIRL0360	Ethyl chloroacetate	105-39-5	ClCH2CO2C2H5	103
QIRL0370	2-Butenal	123-73-9	CH3CHCHCHO	158
QIRL0380	Ethyl chloroformate	541-41-3	ClCO2C2H5	209
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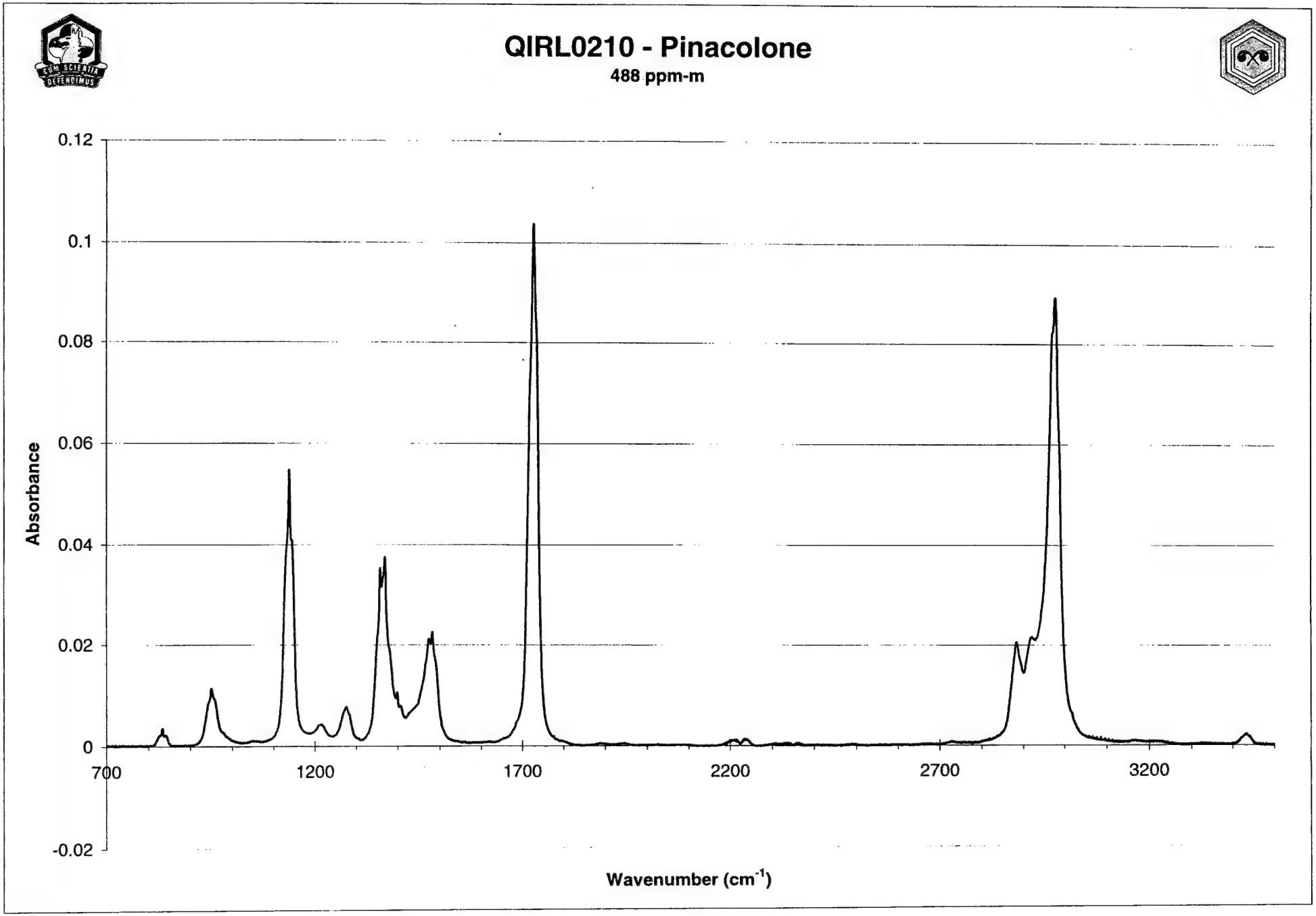
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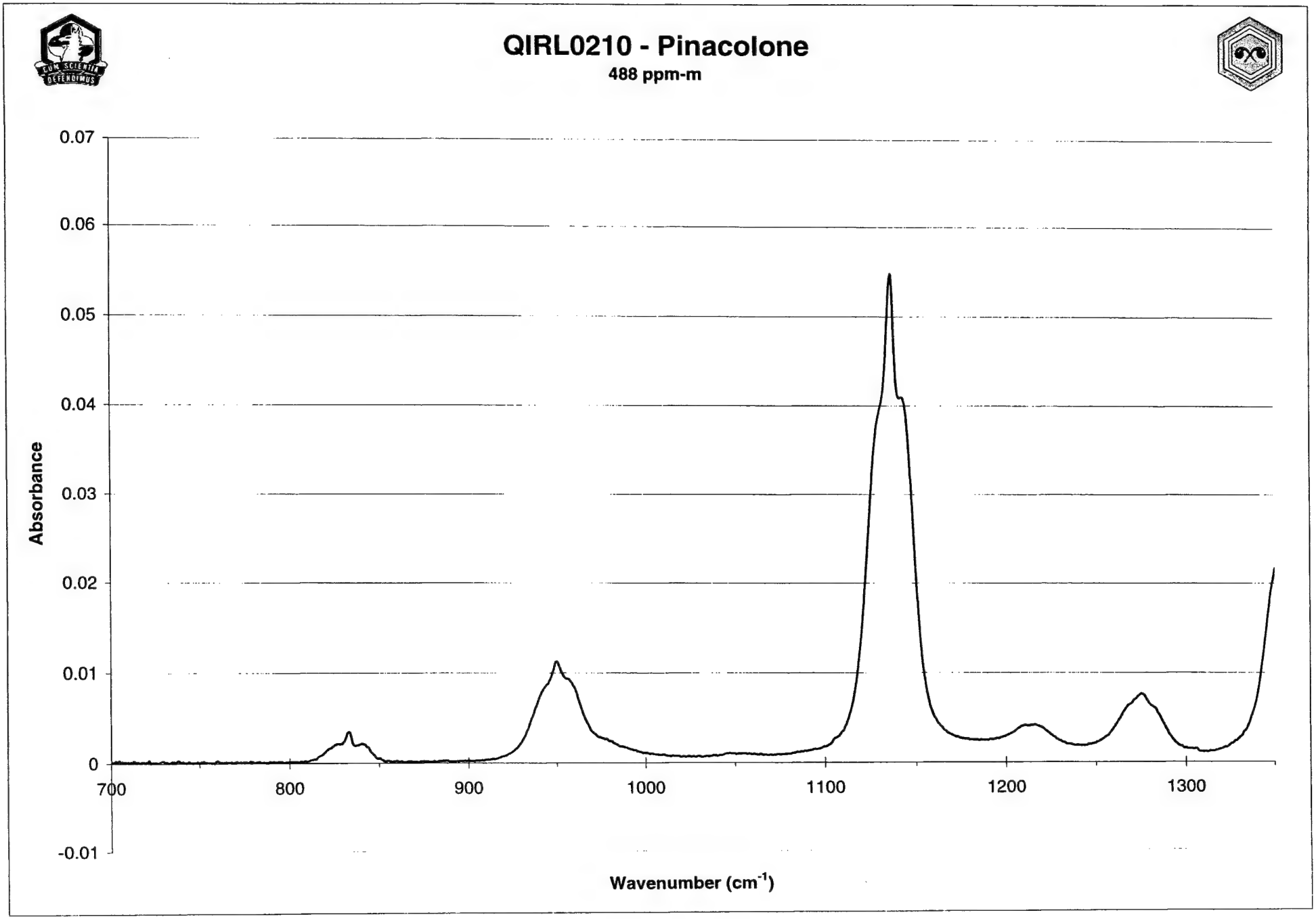
3200
Wavenumber (cm-1)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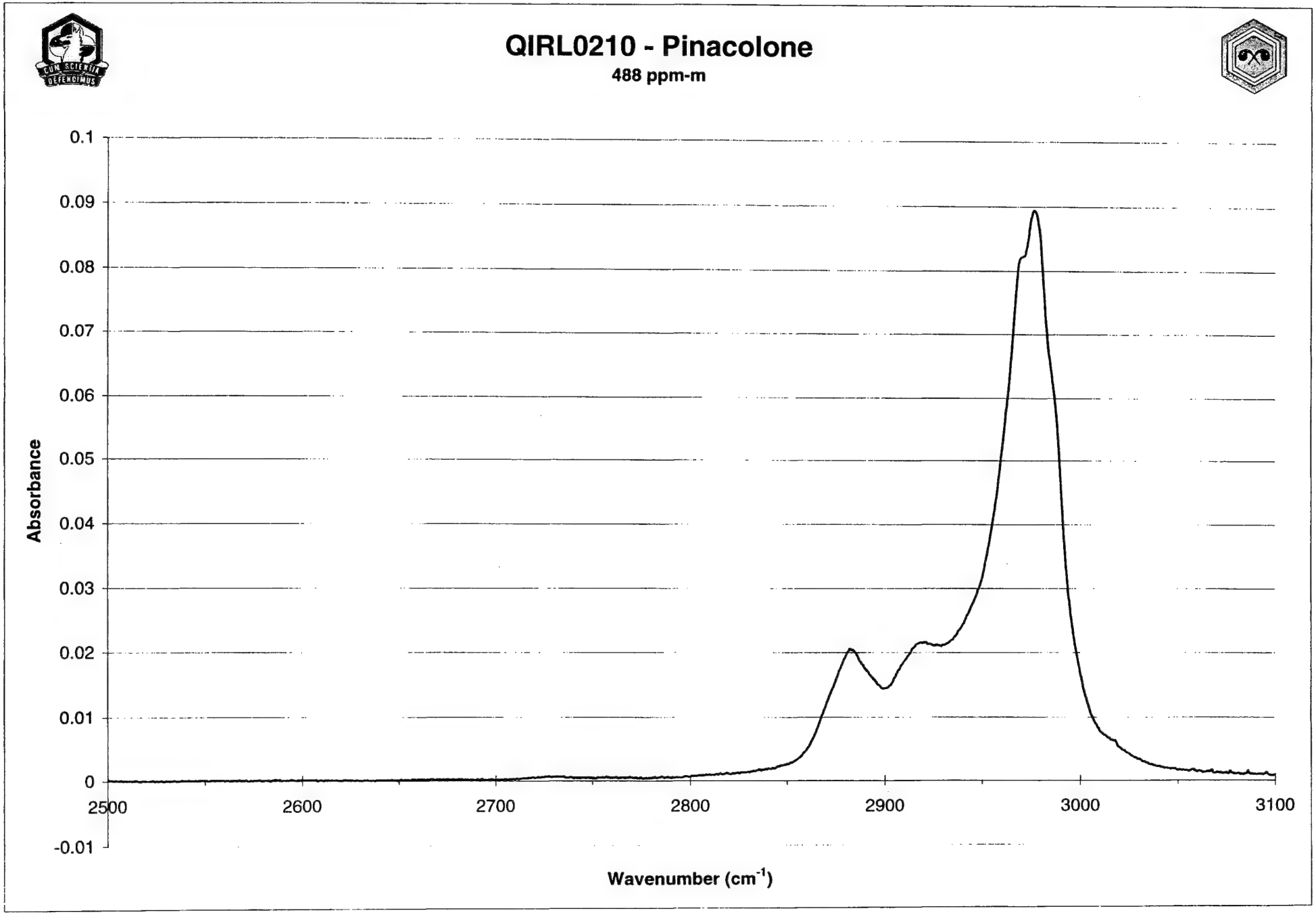
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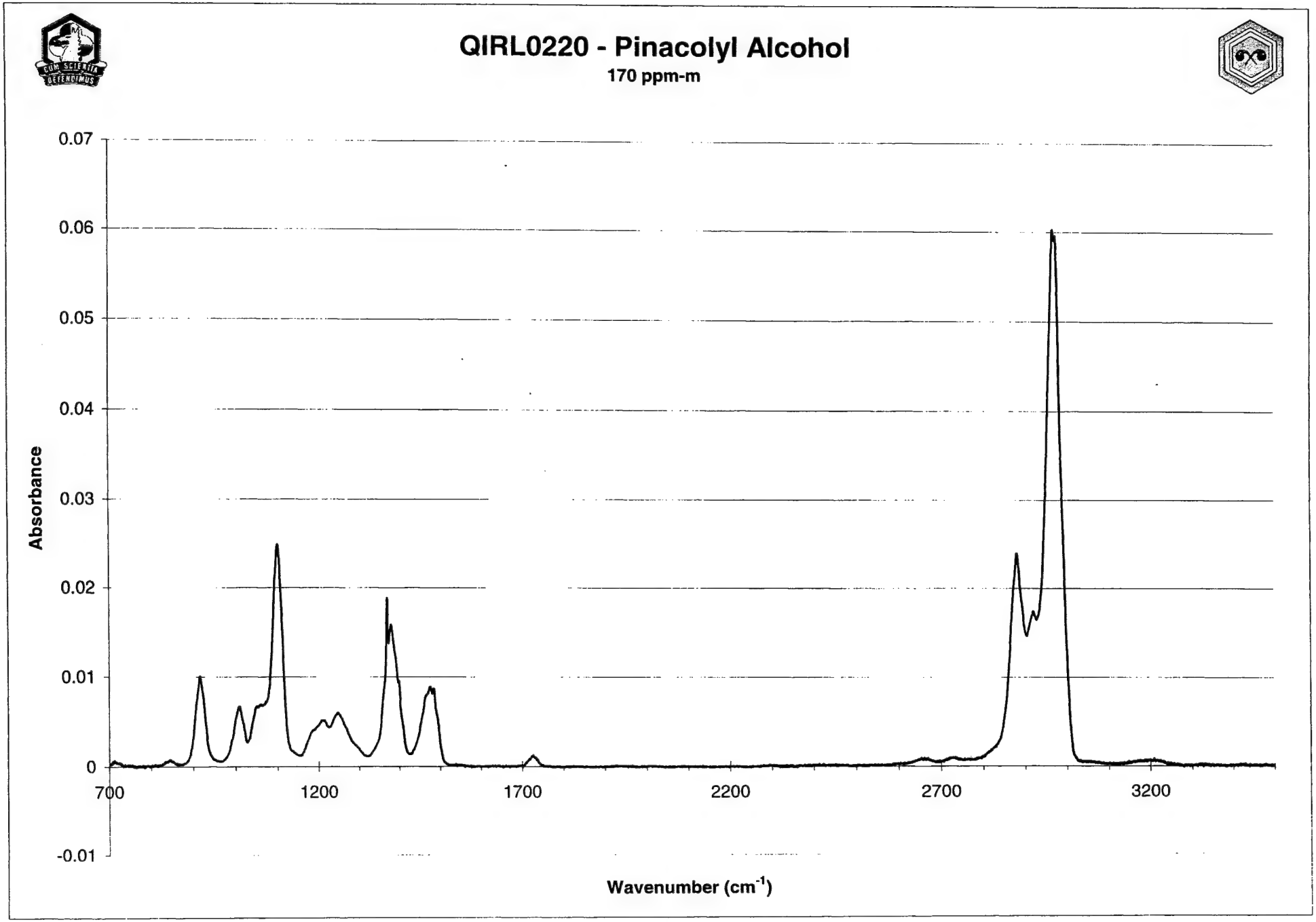
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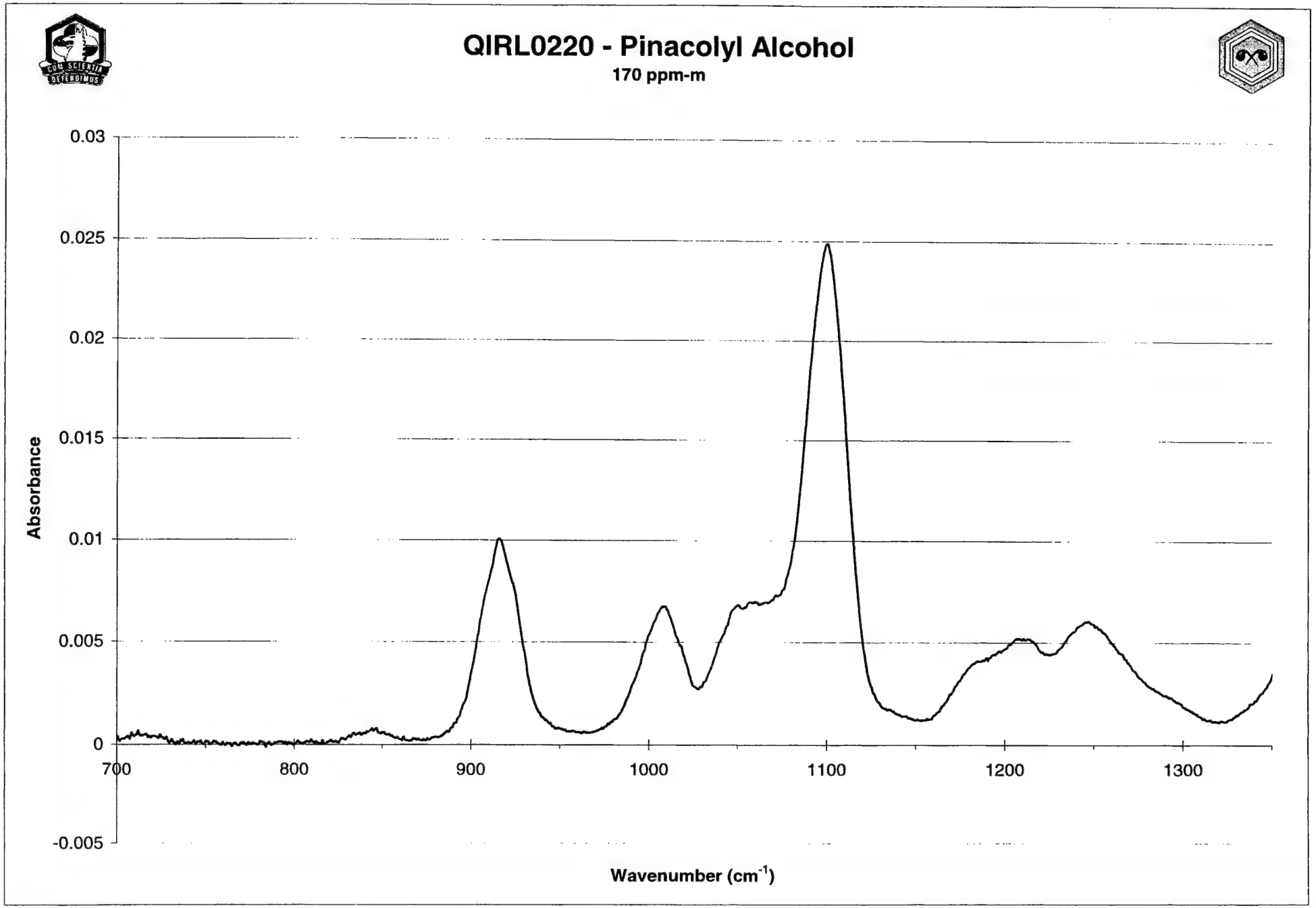
Wavenumber (cm-1)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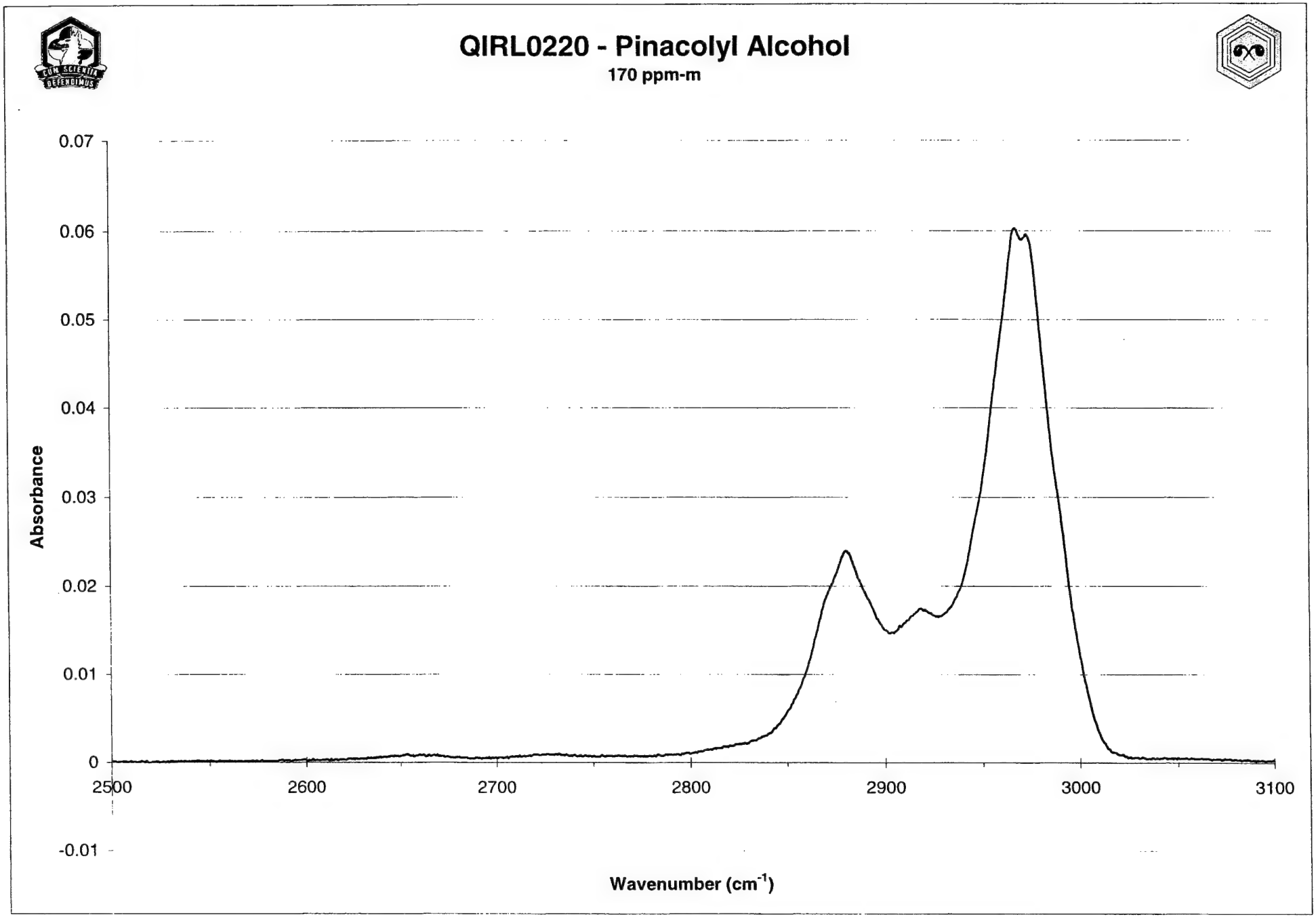
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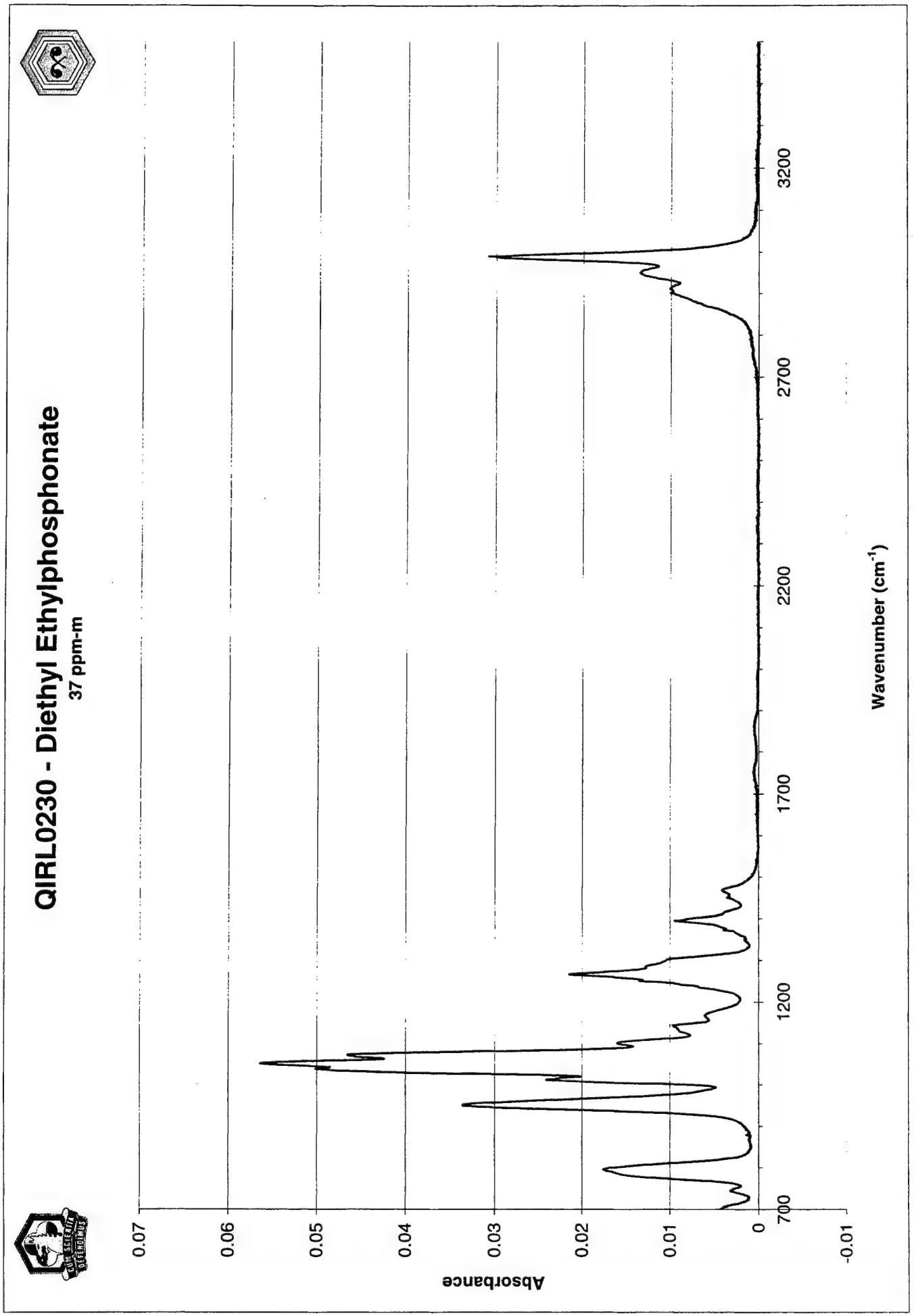
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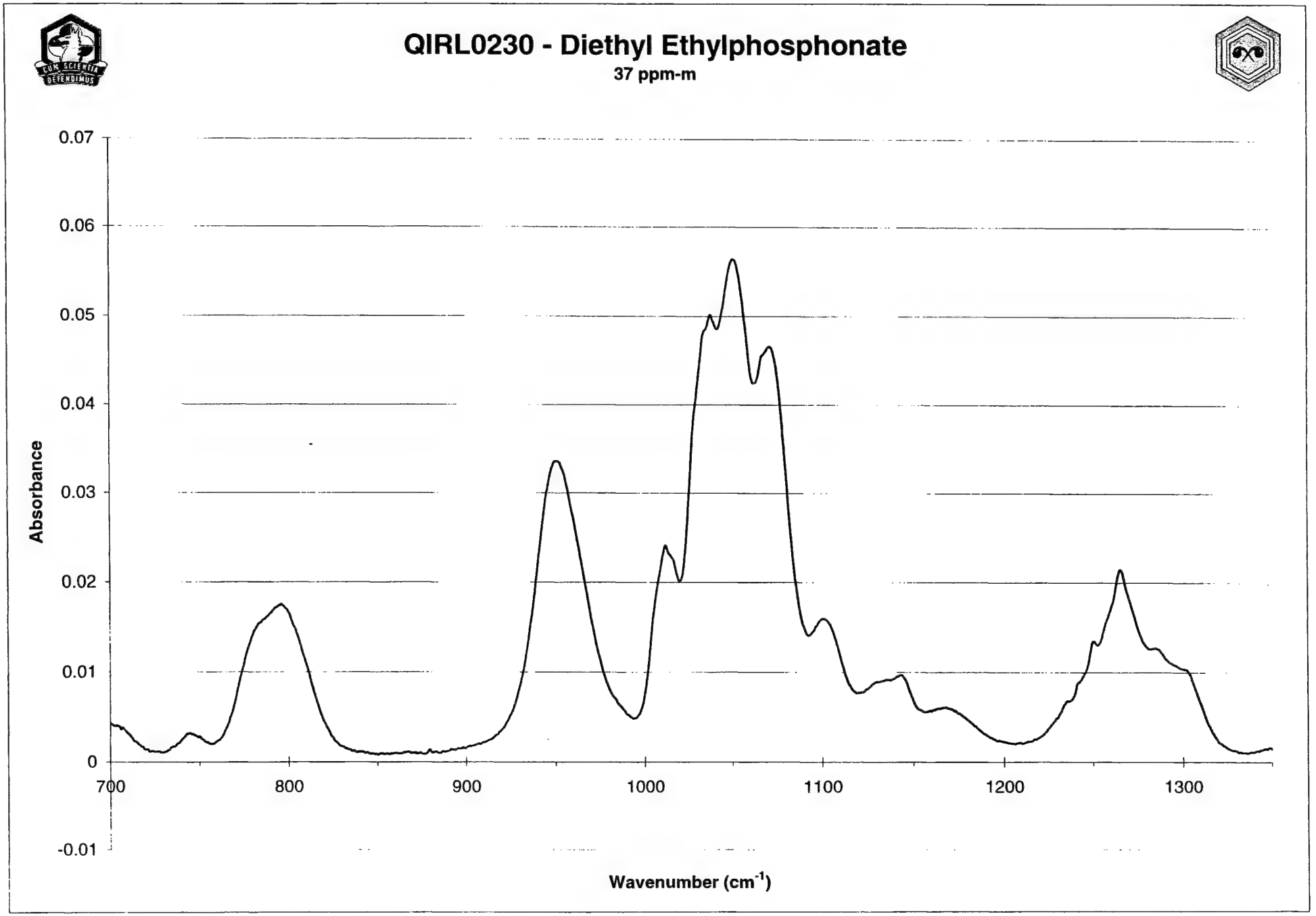
Wavenumber (cm-1)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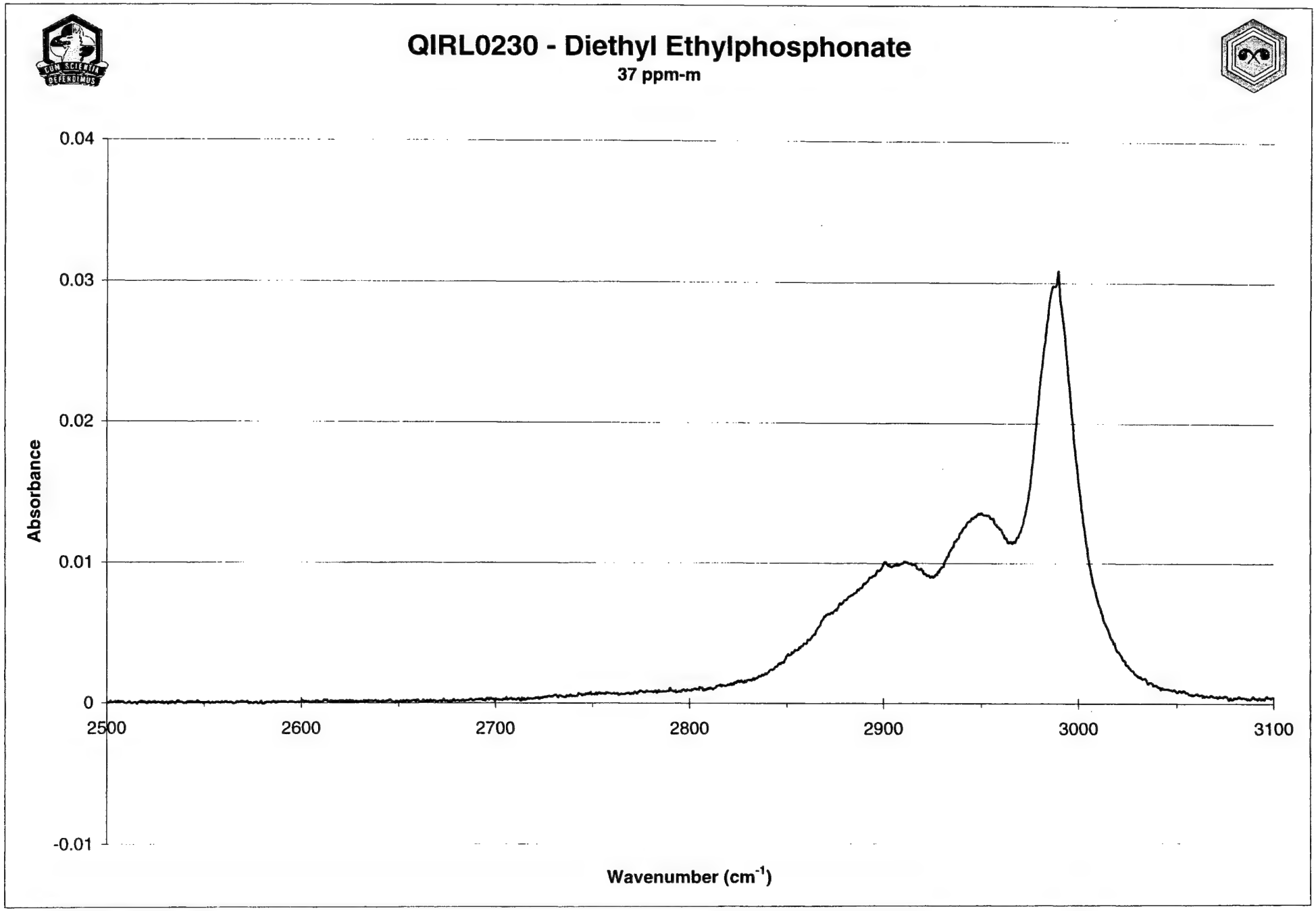
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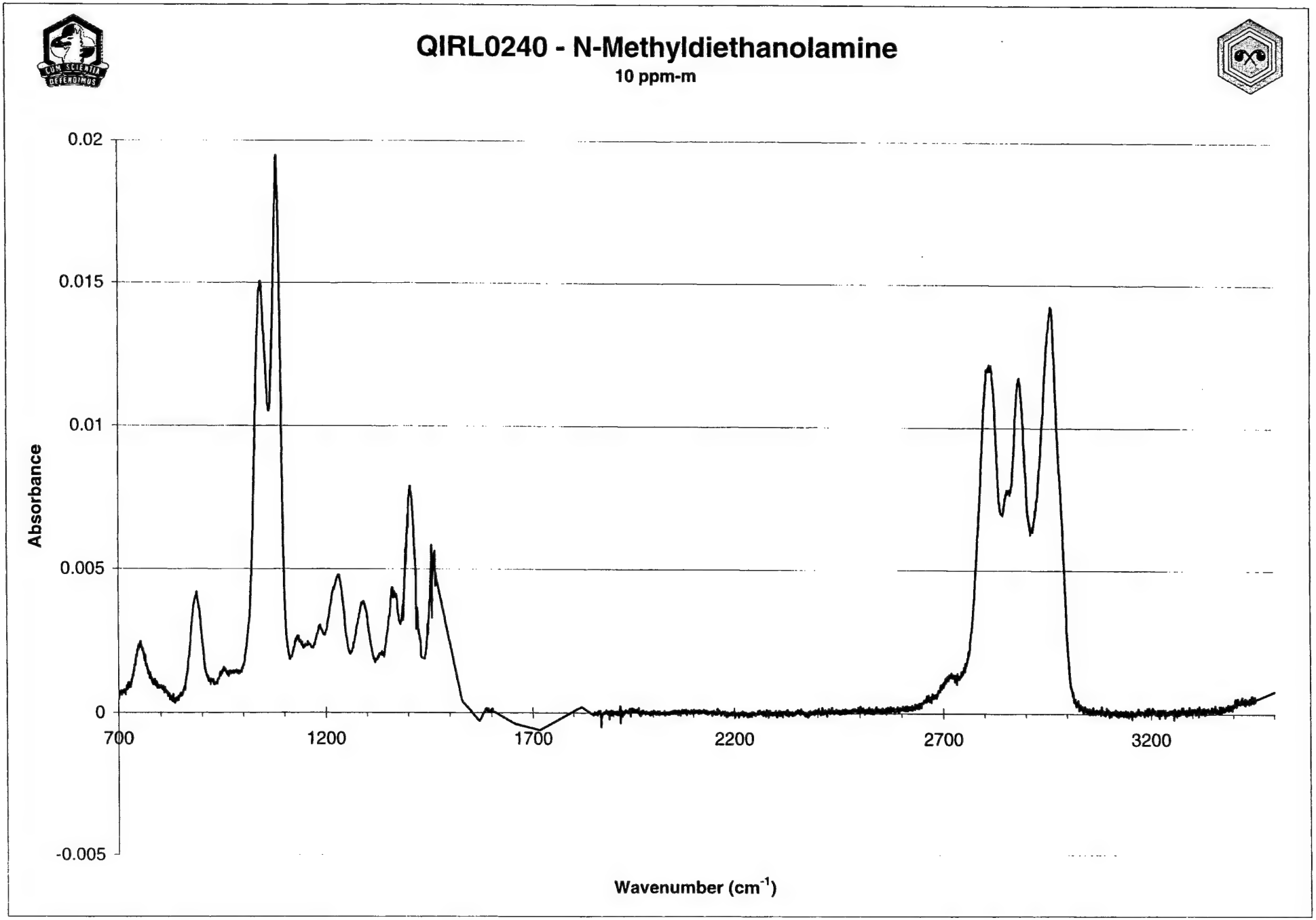
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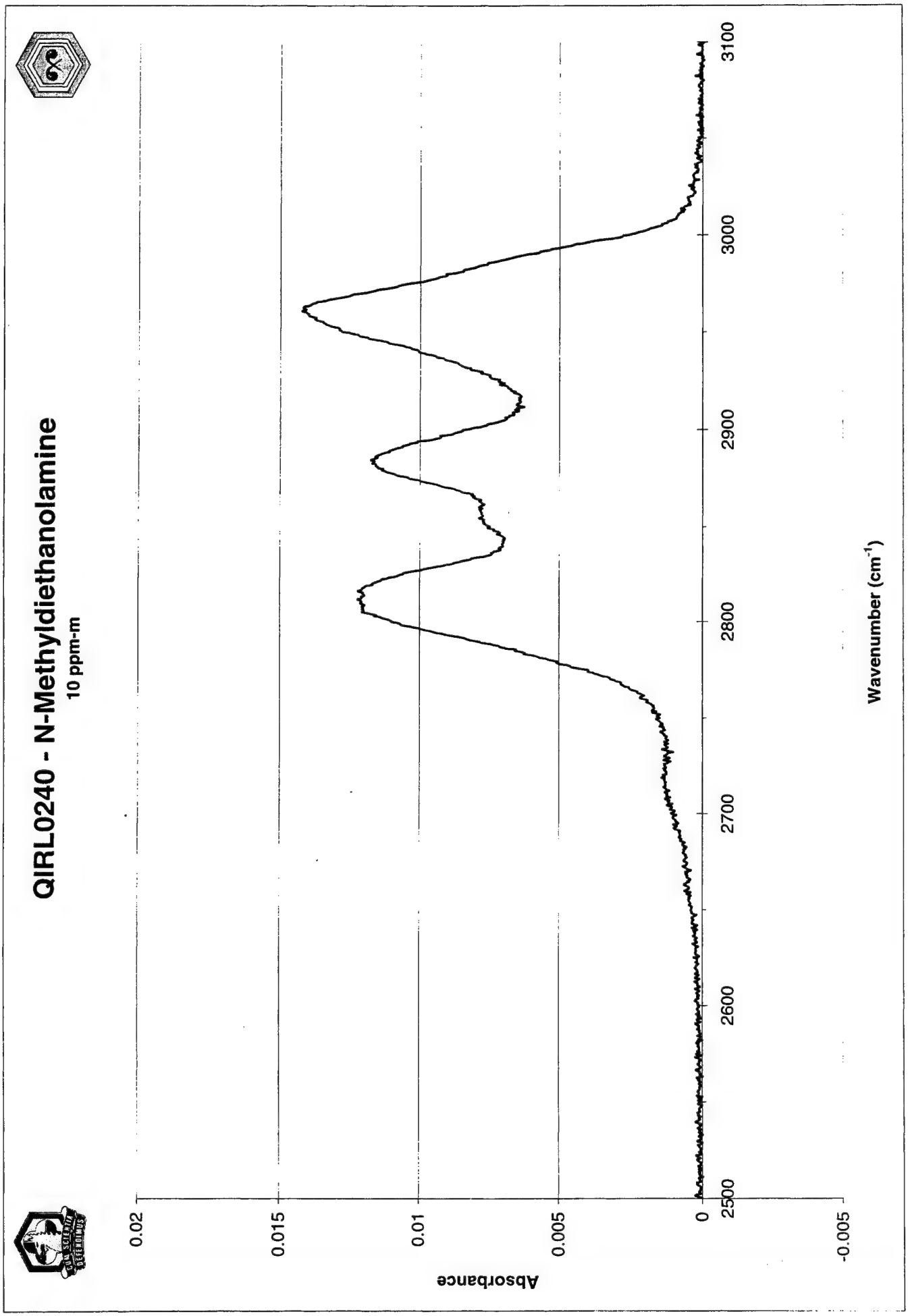
Wavenumber (cm-1)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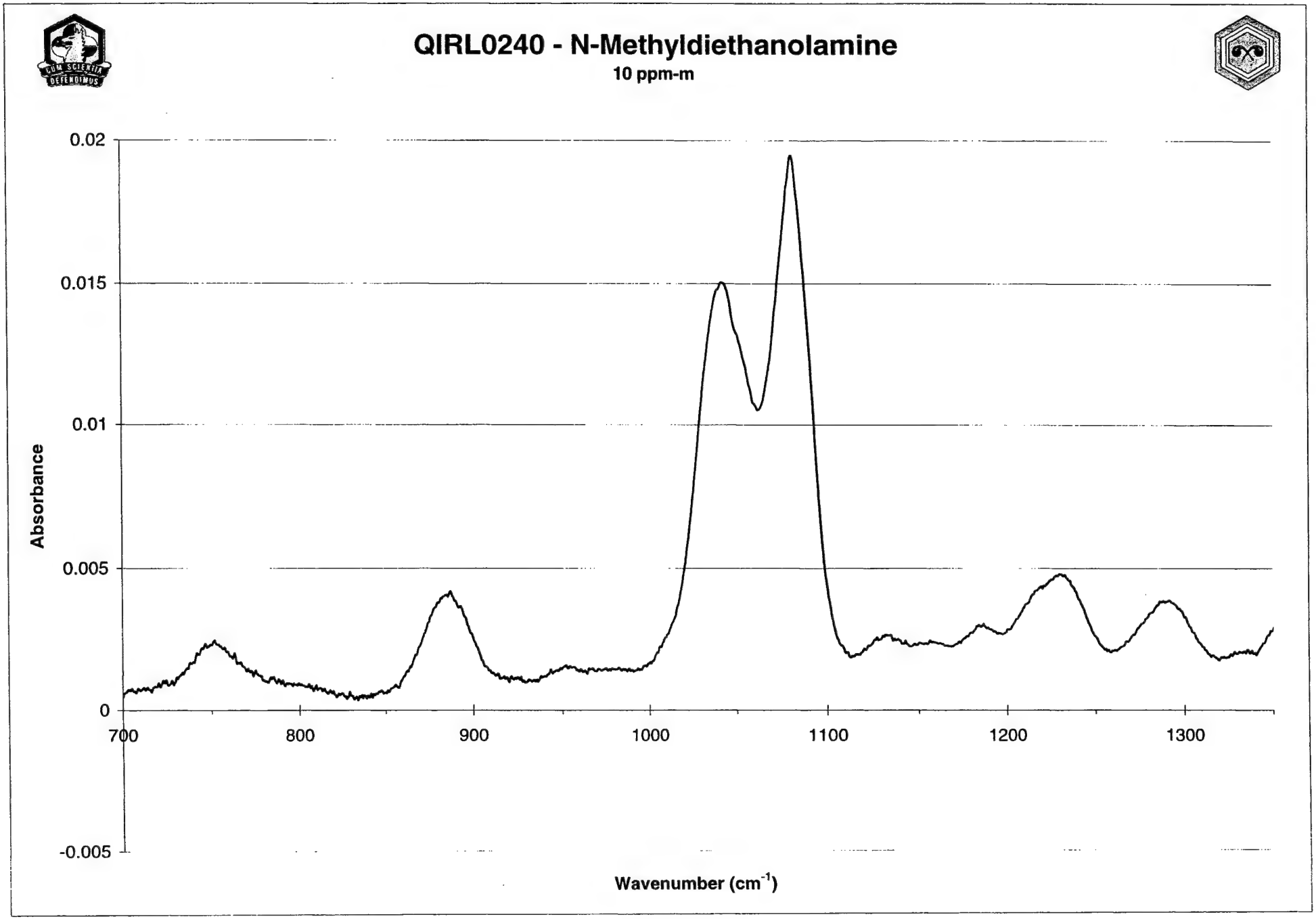
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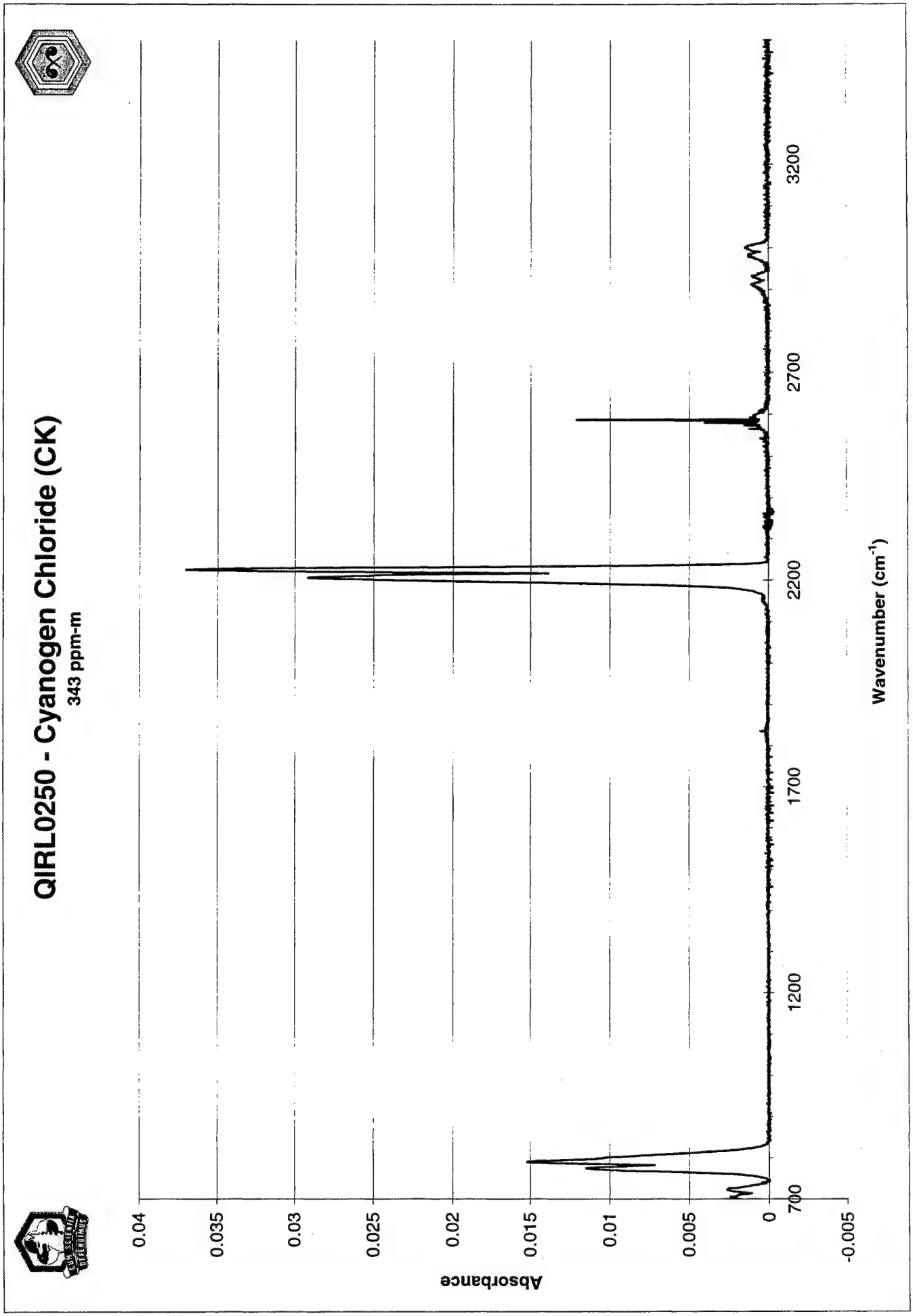
Wavenumber (cm-1)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Absorbance
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0.025
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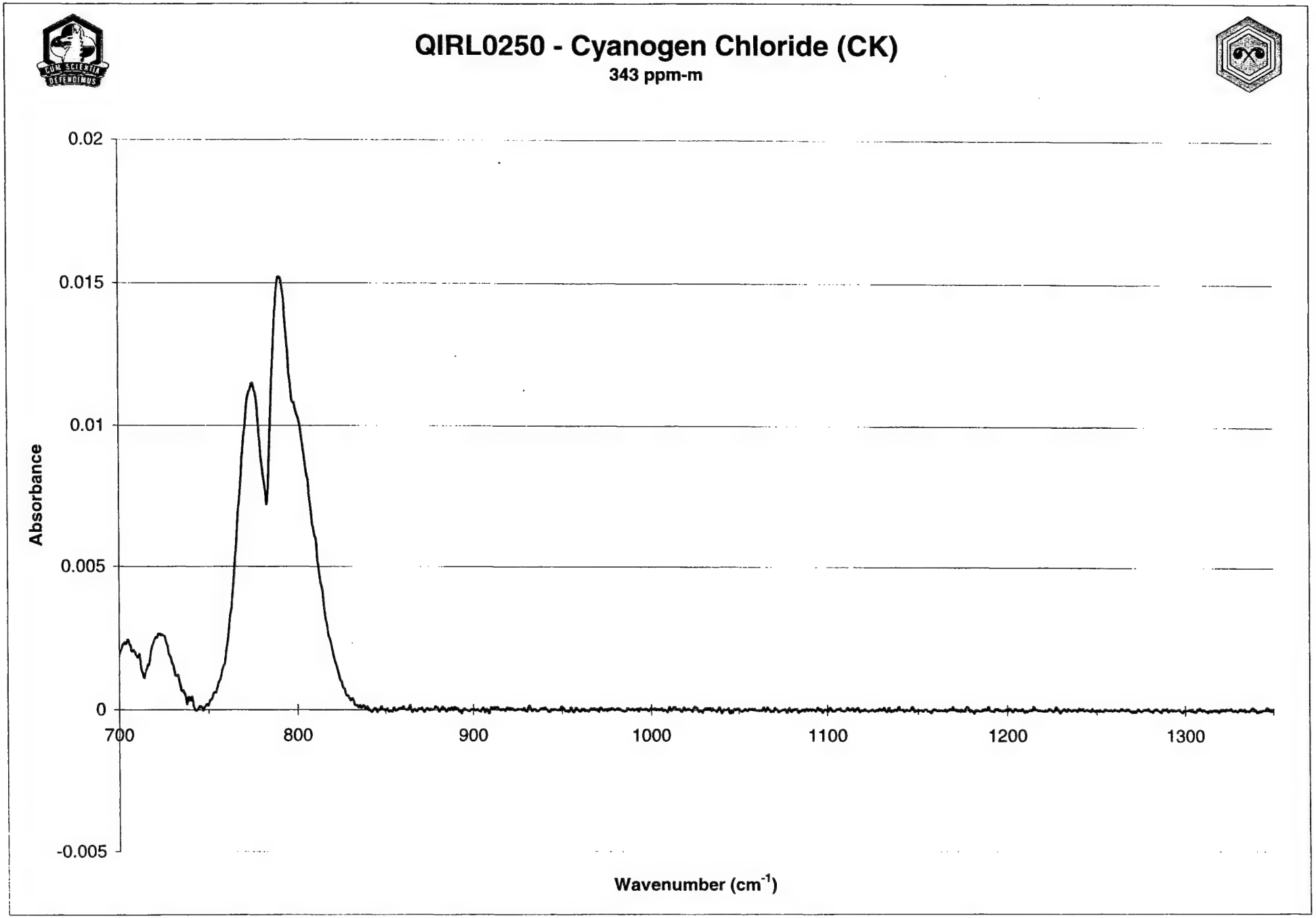
Wavenumber (cm-1)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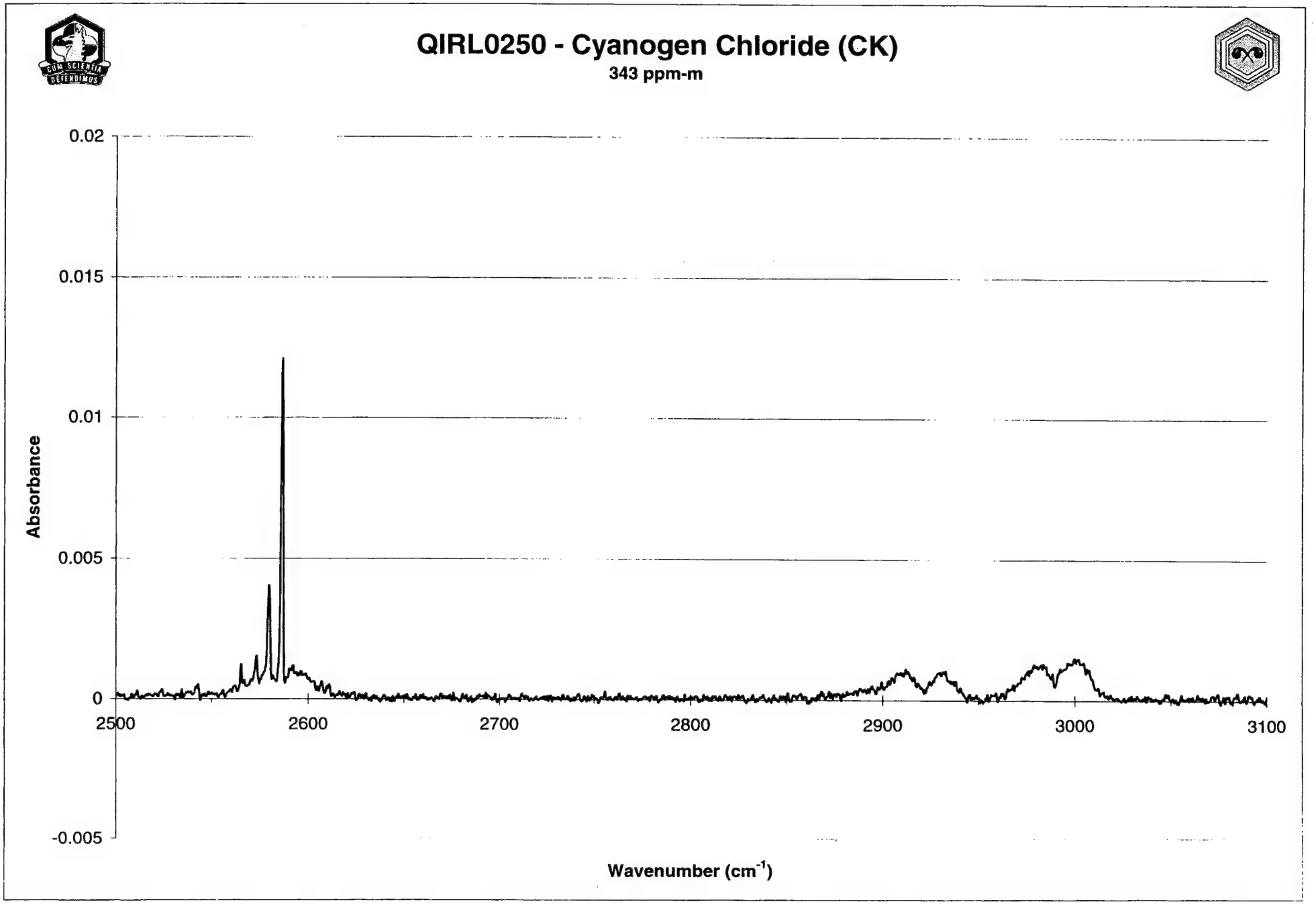
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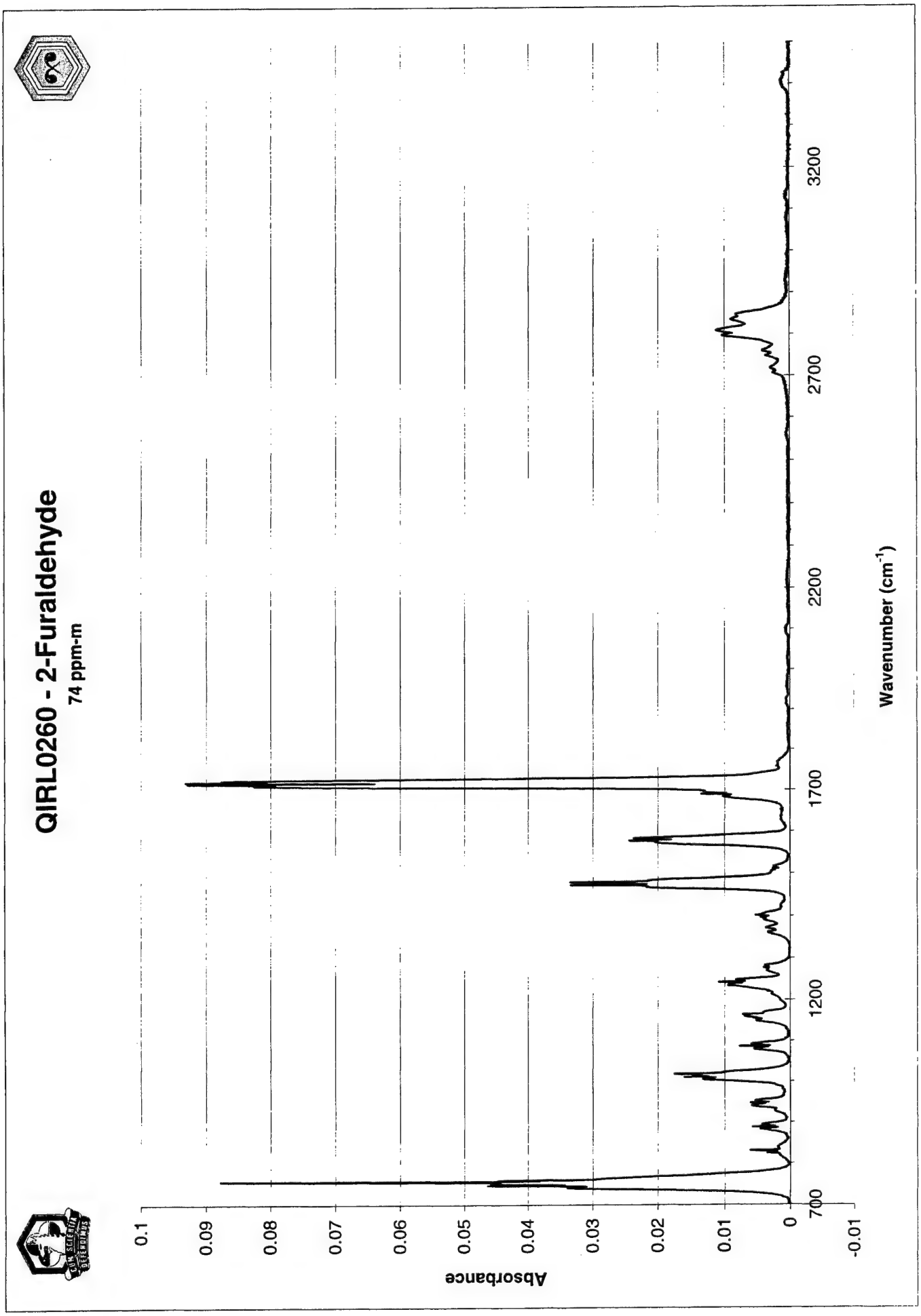
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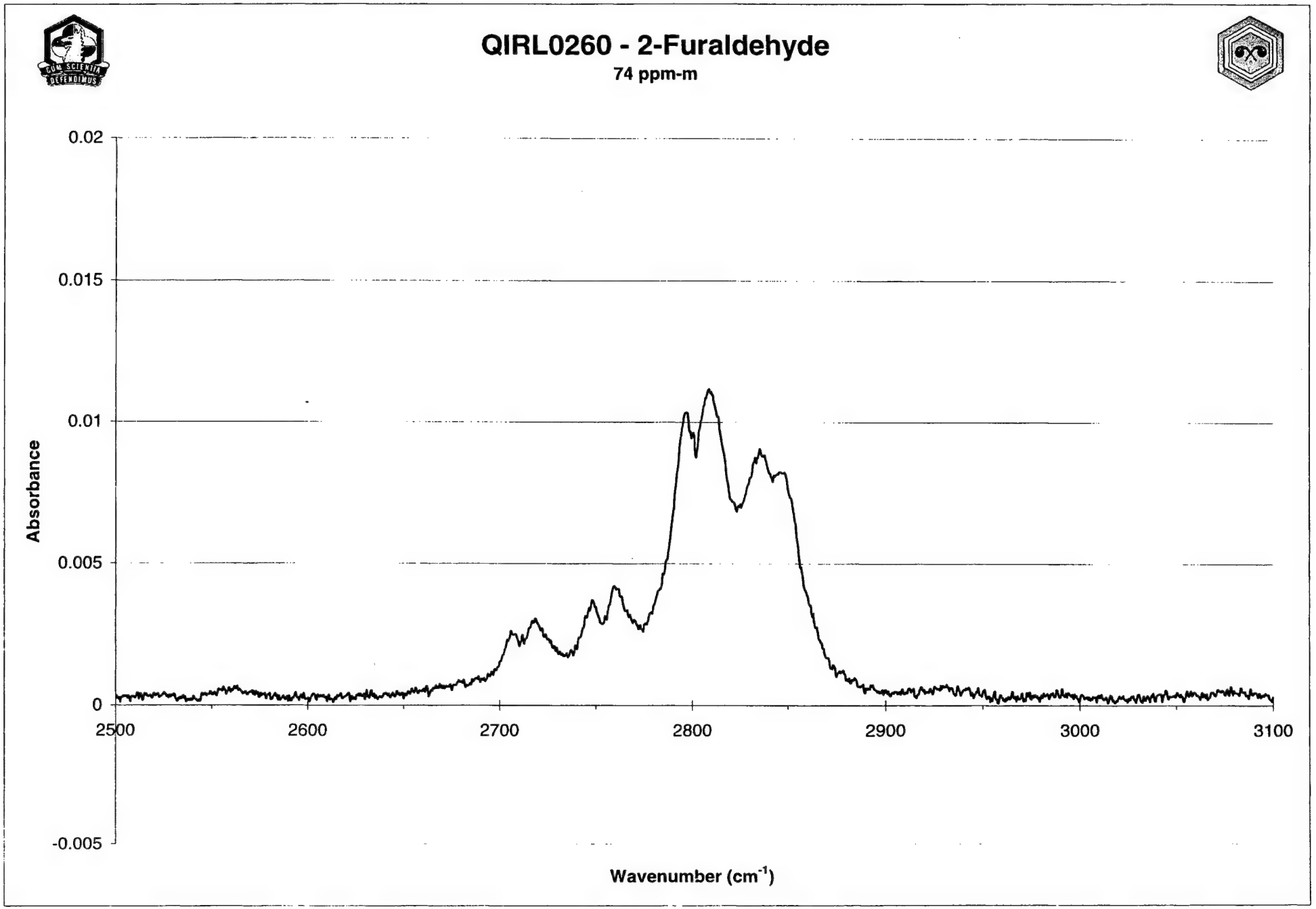
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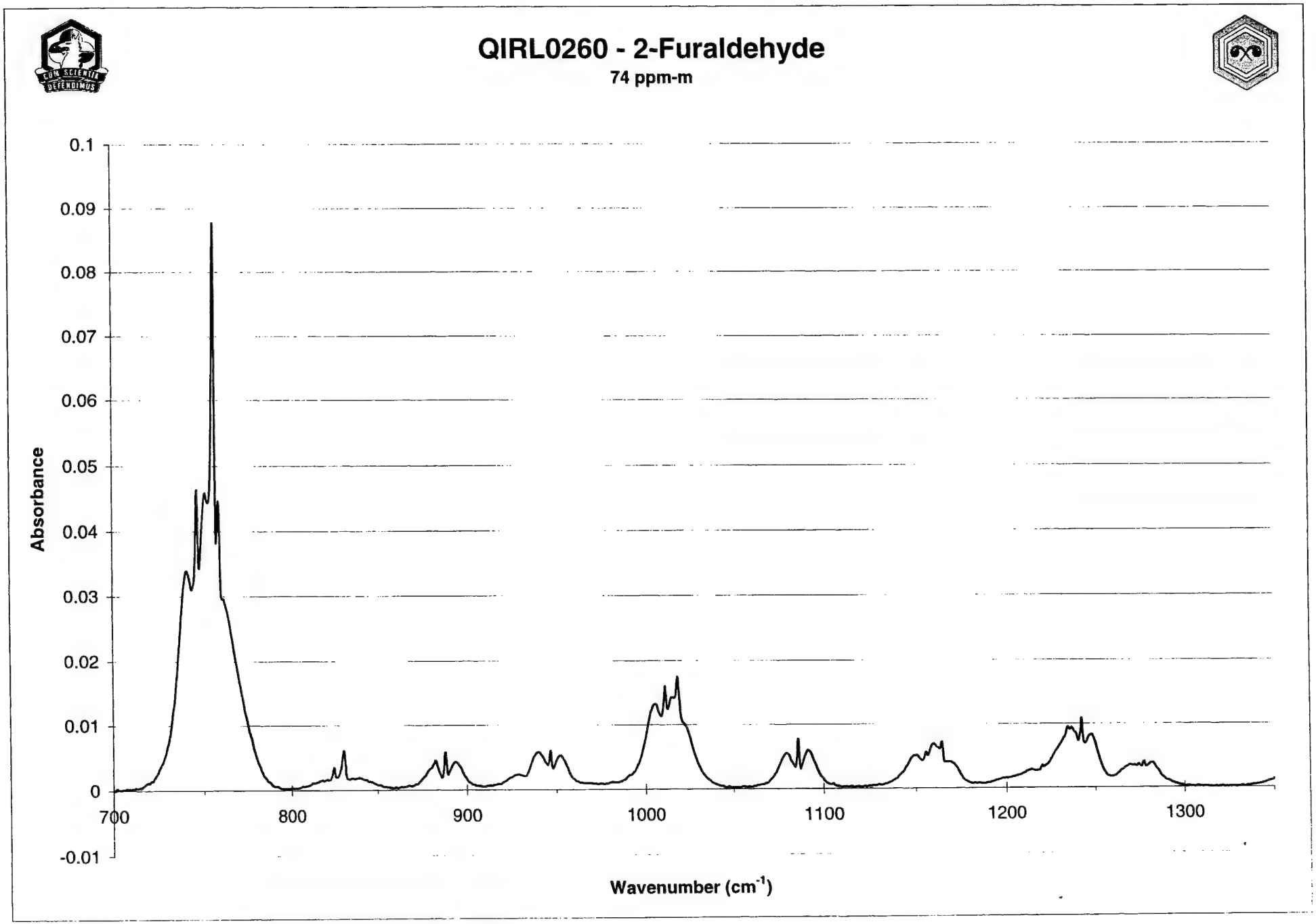
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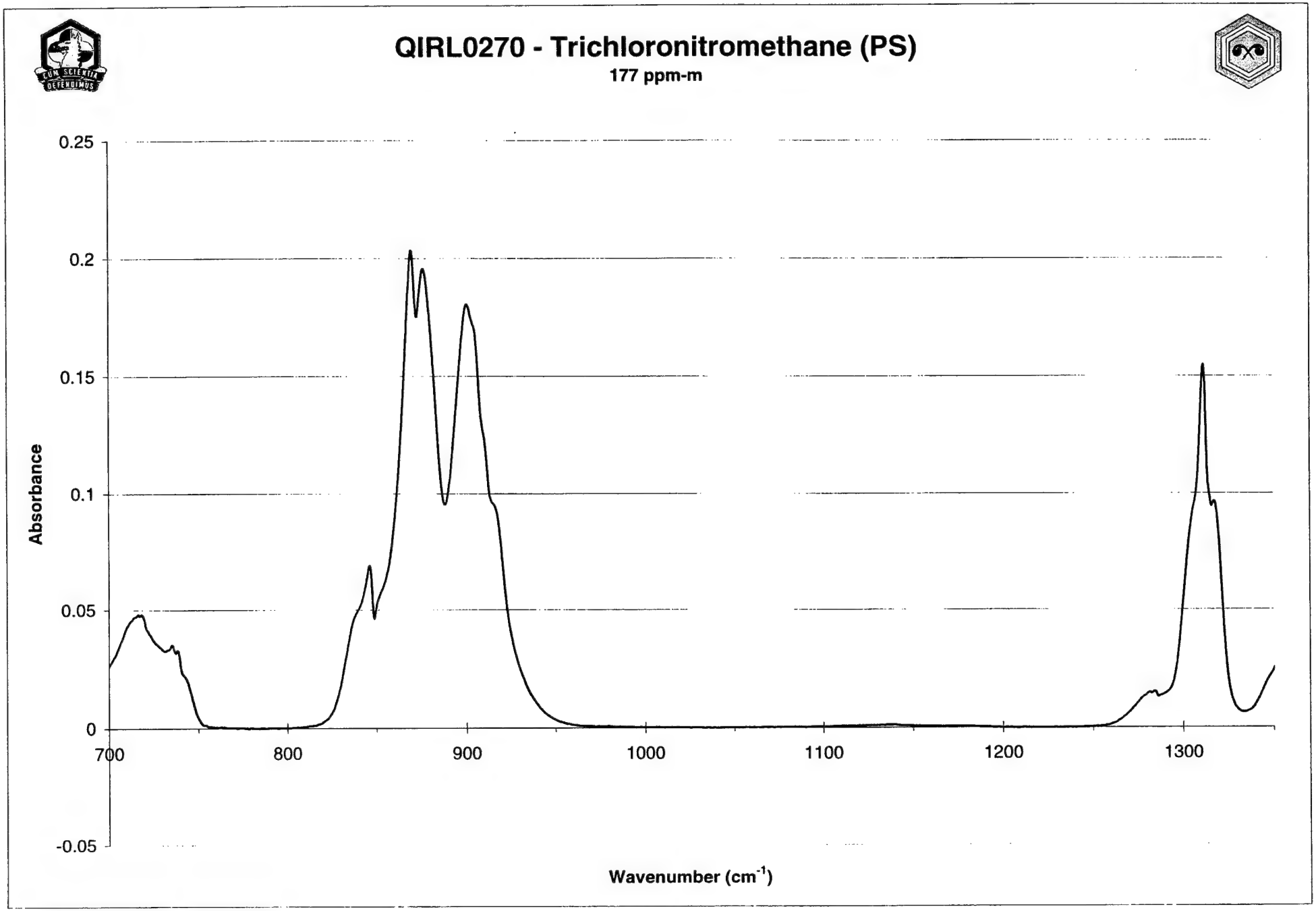
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Absorbance
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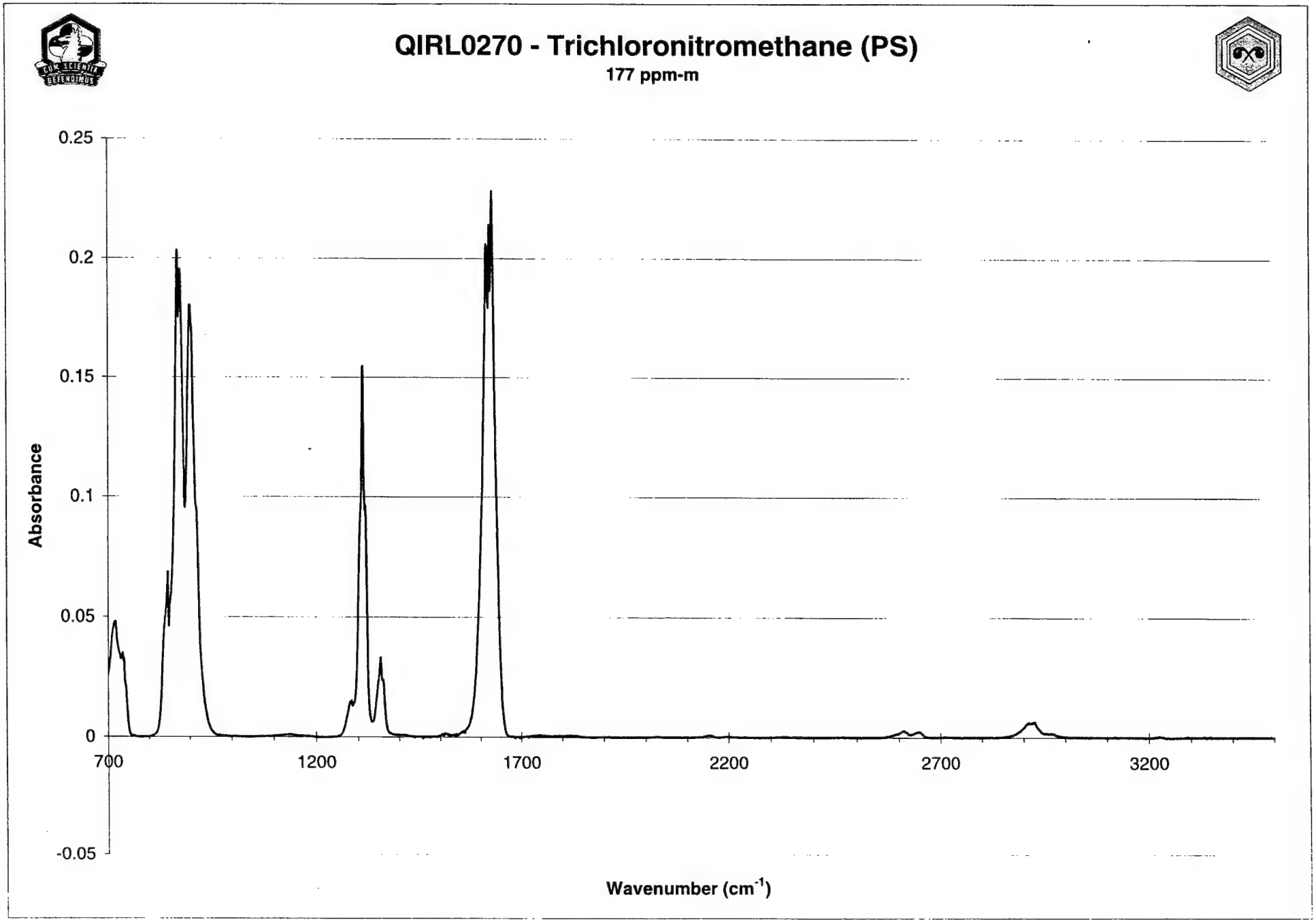
Wavenumber (cm-1)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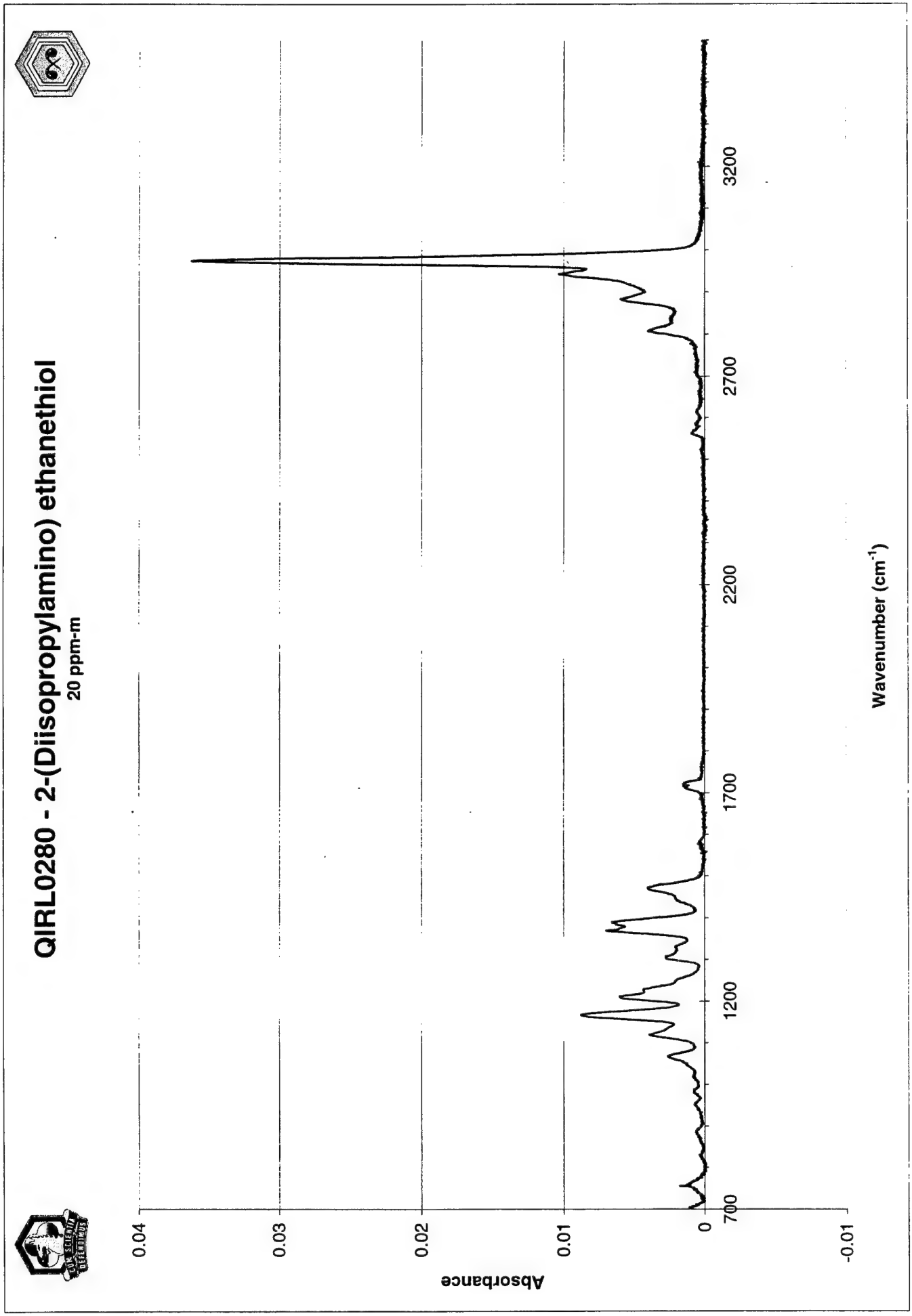
Wavenumber (cm-1)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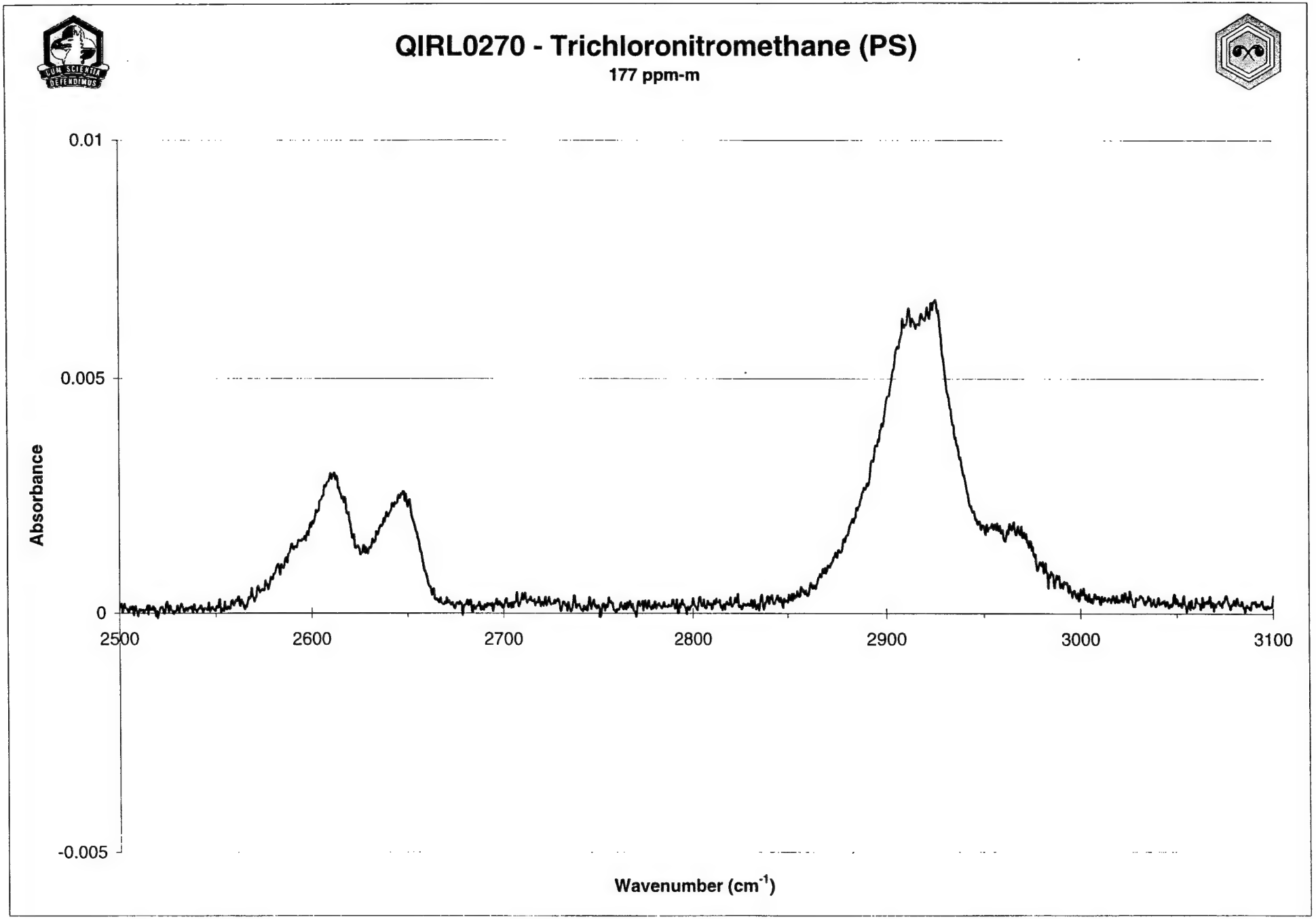
Wavenumber (cm-1)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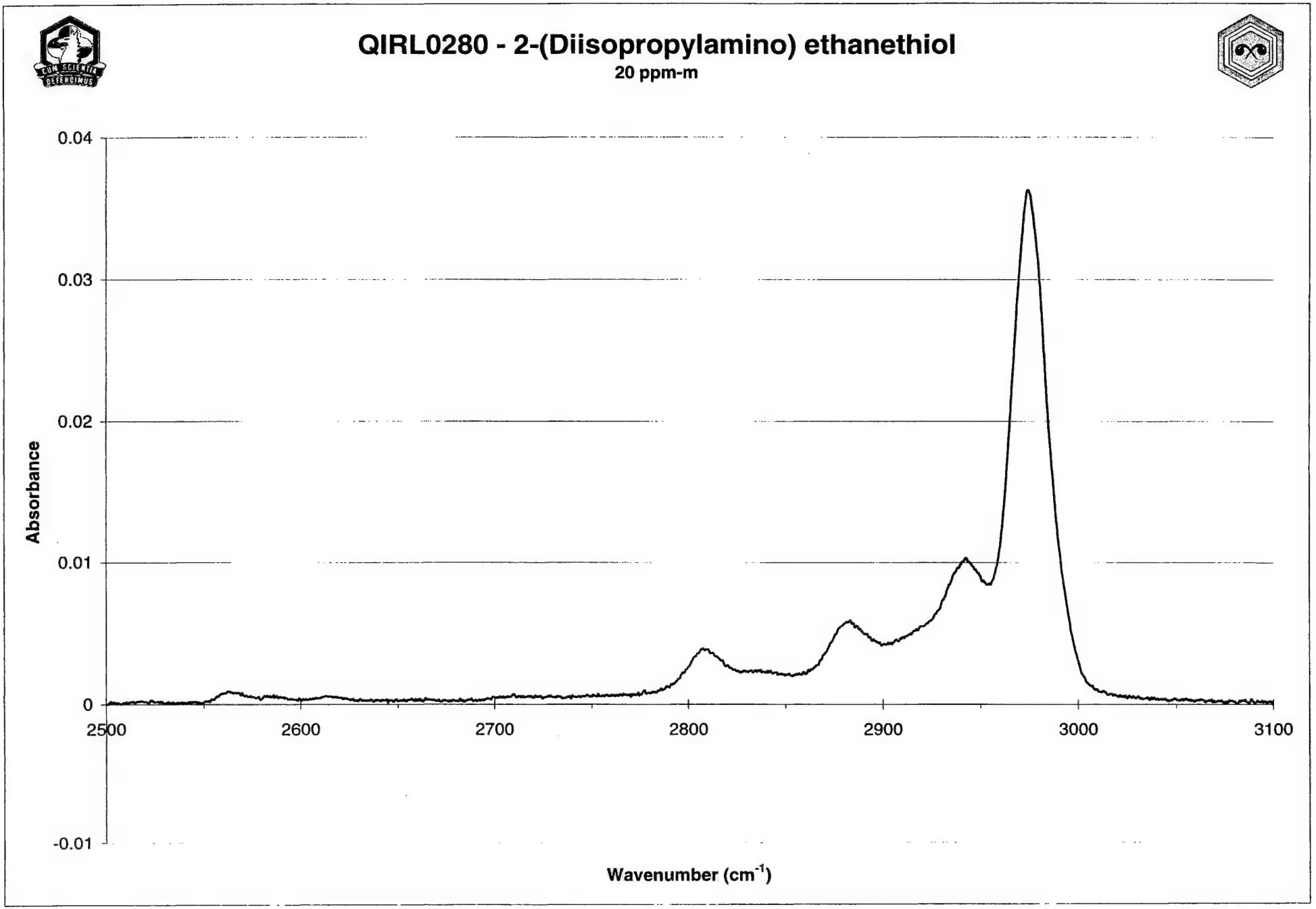
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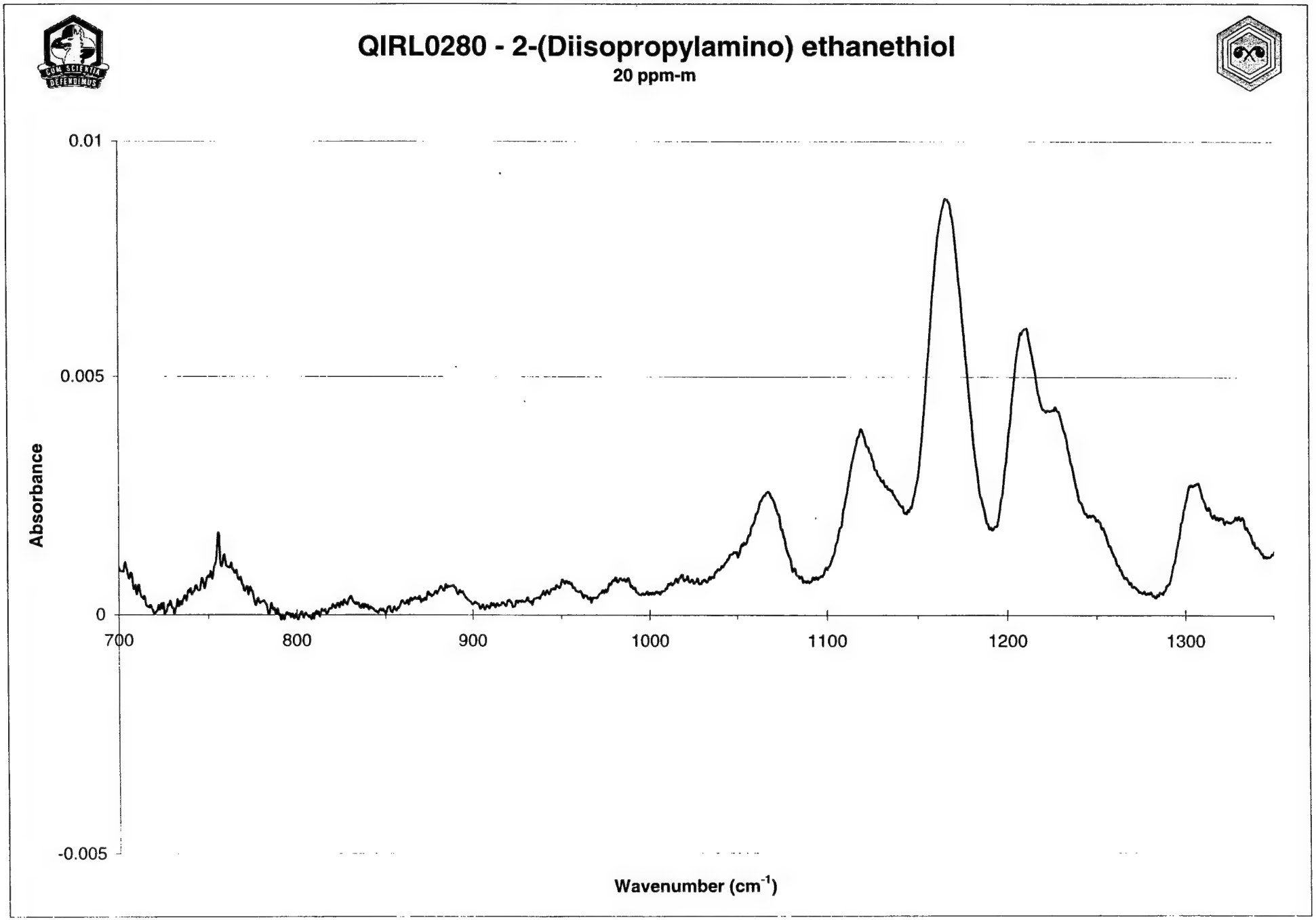
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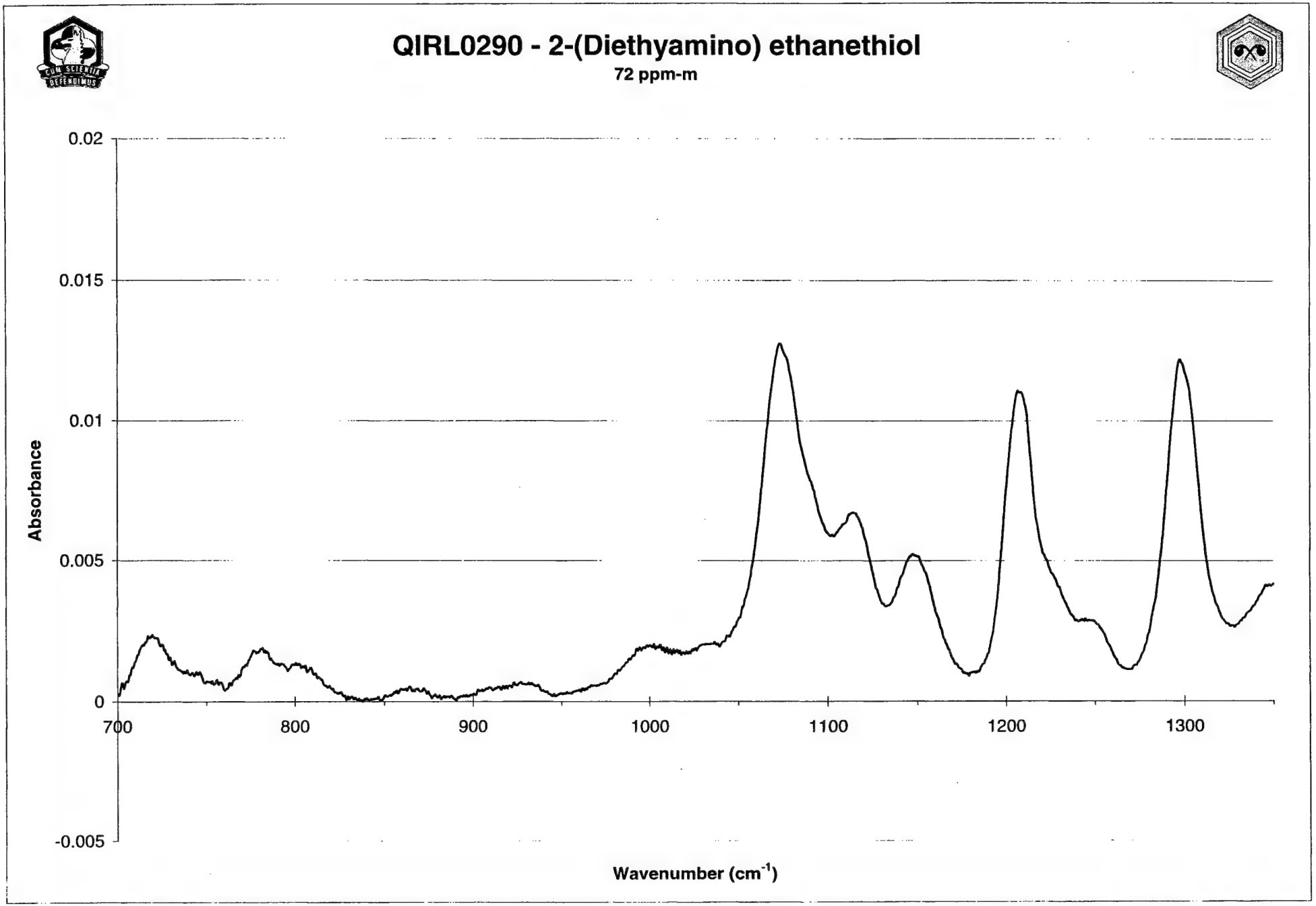
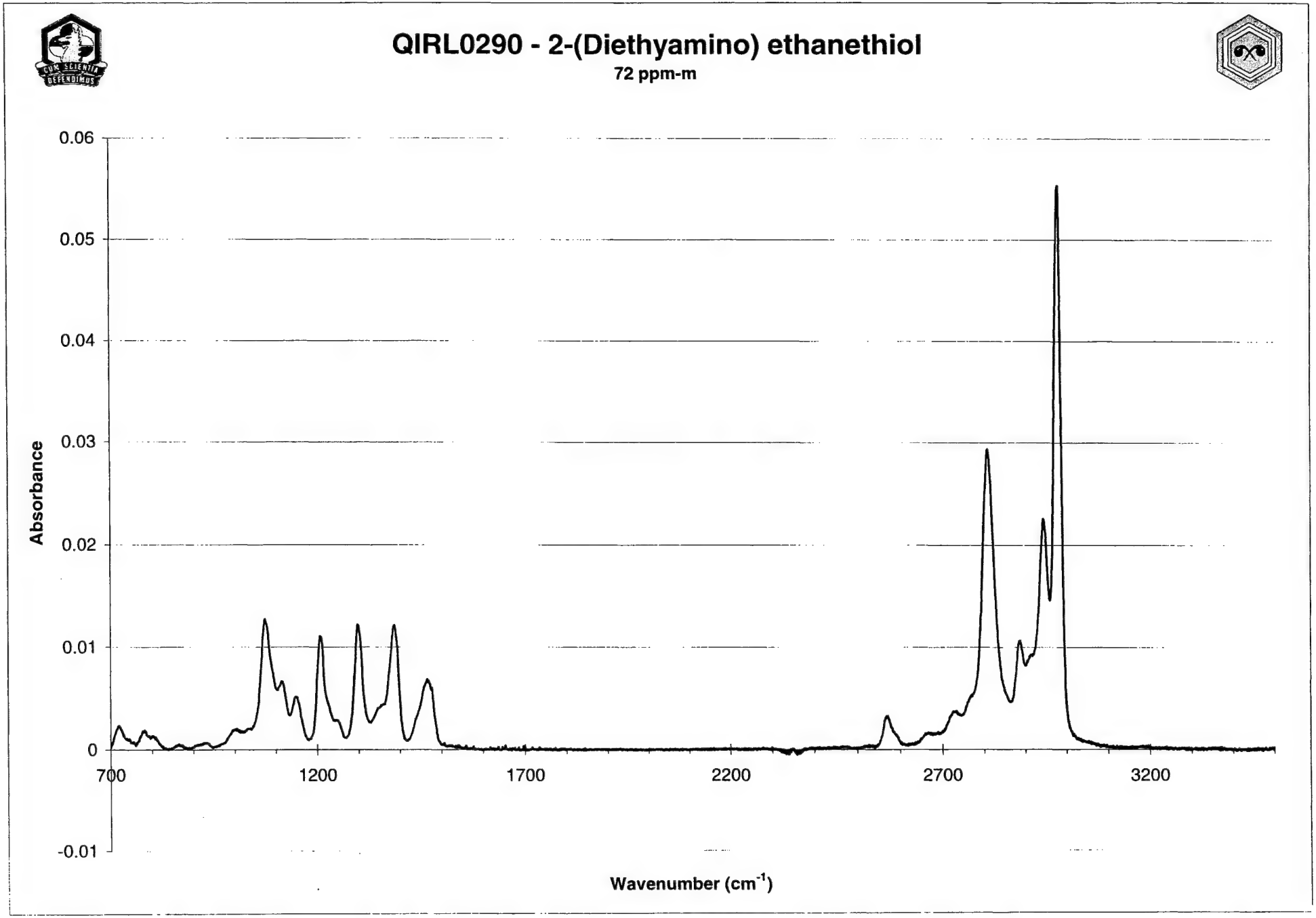
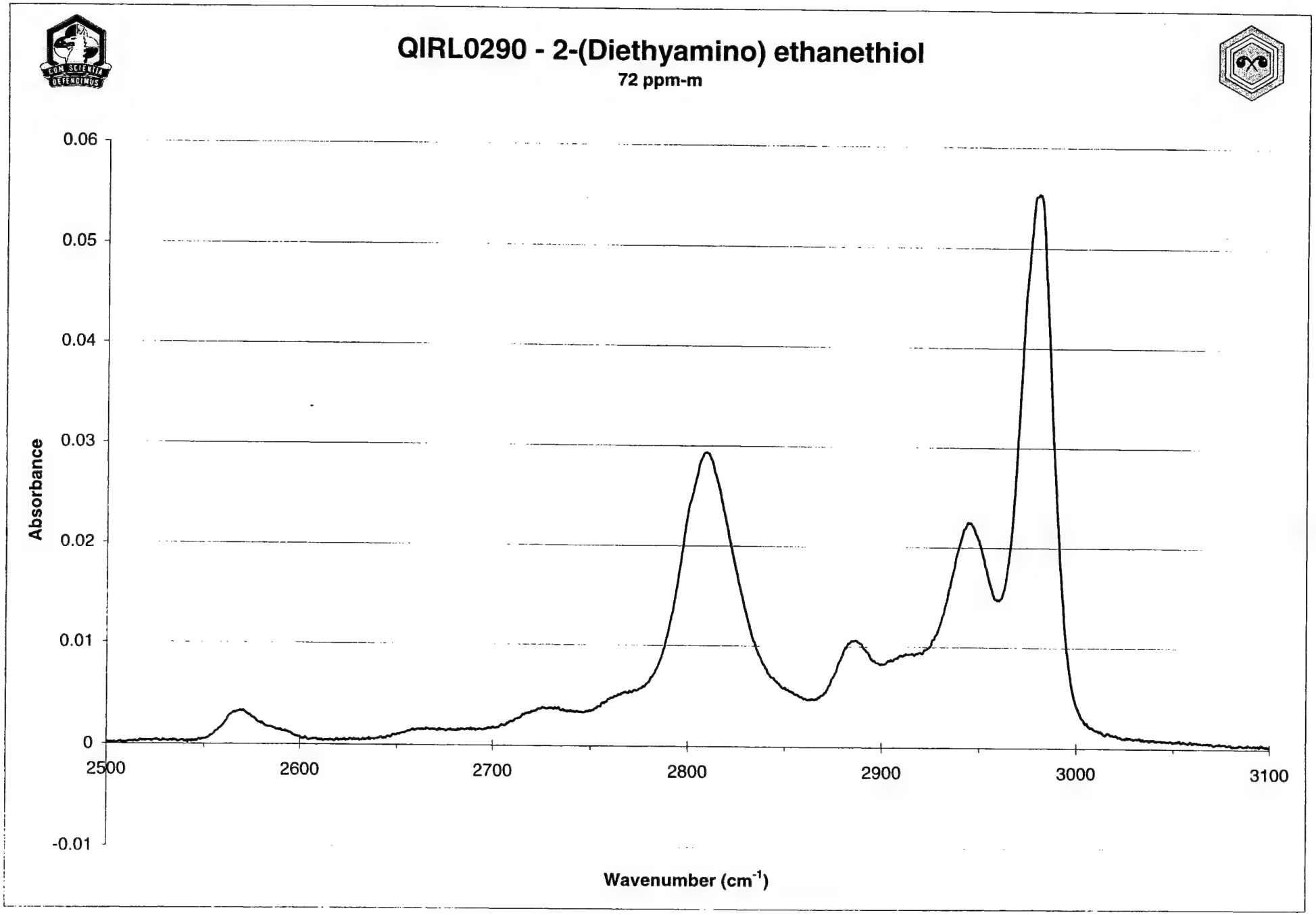
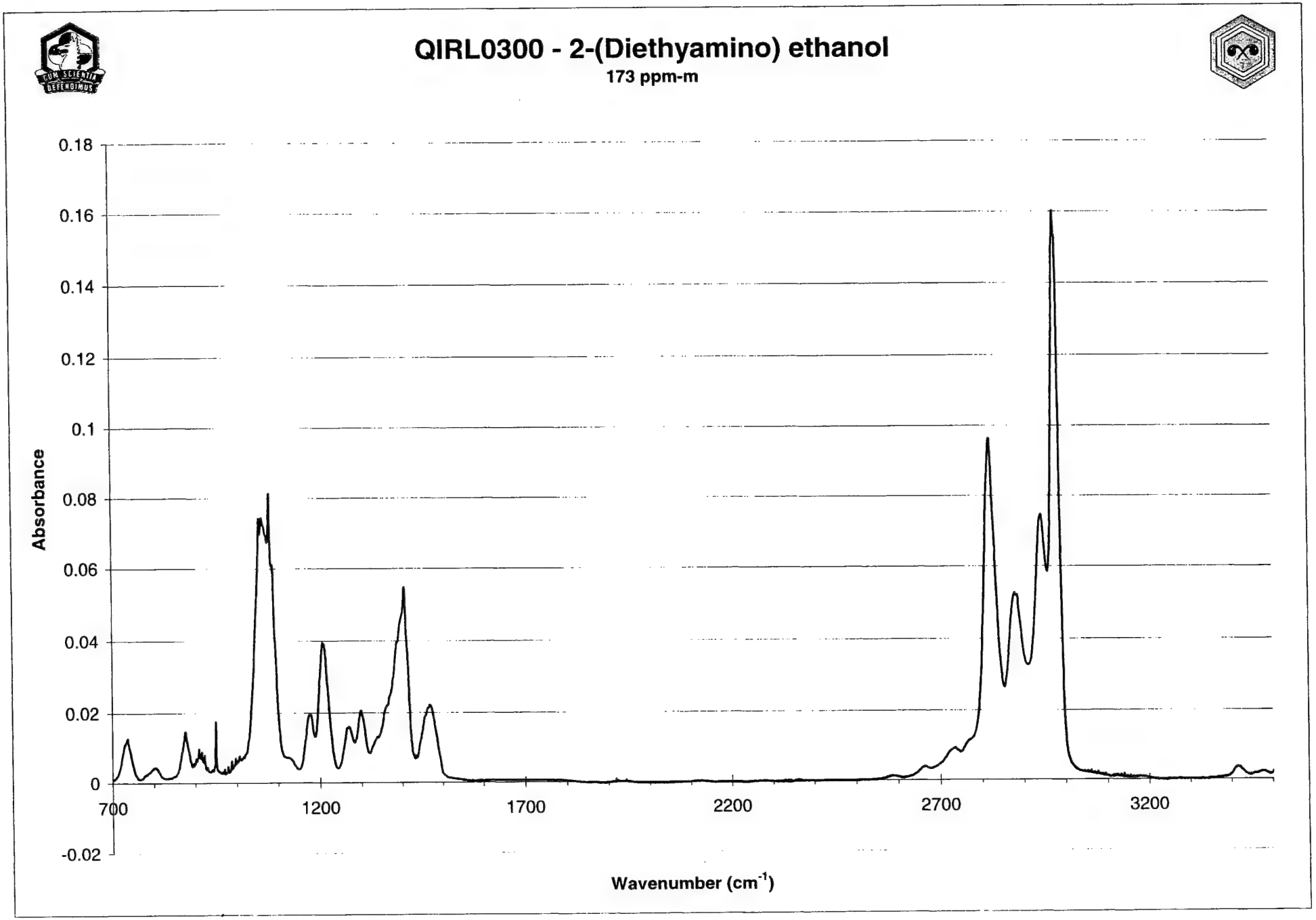
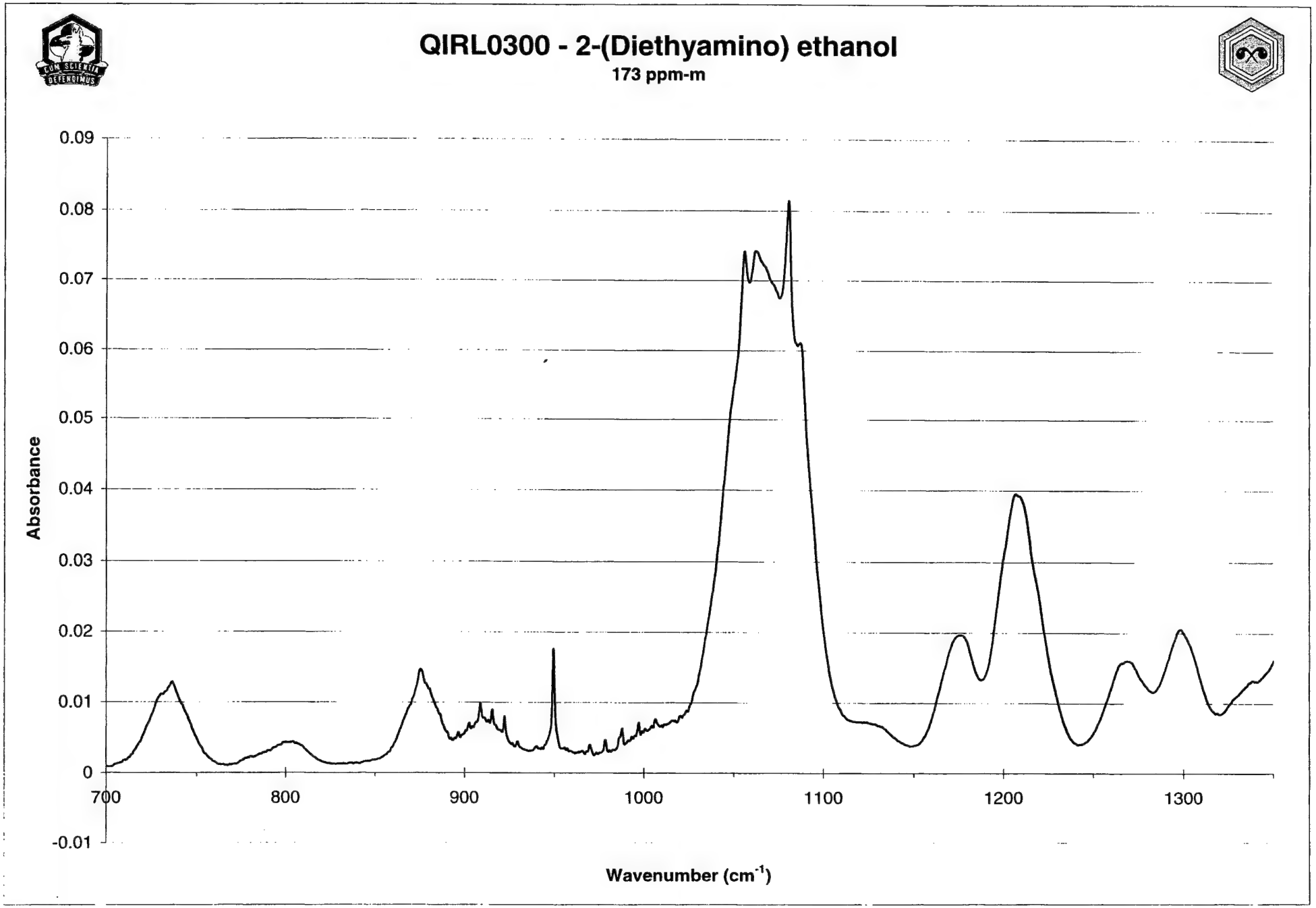
1300
Wavenumber (cm-1)

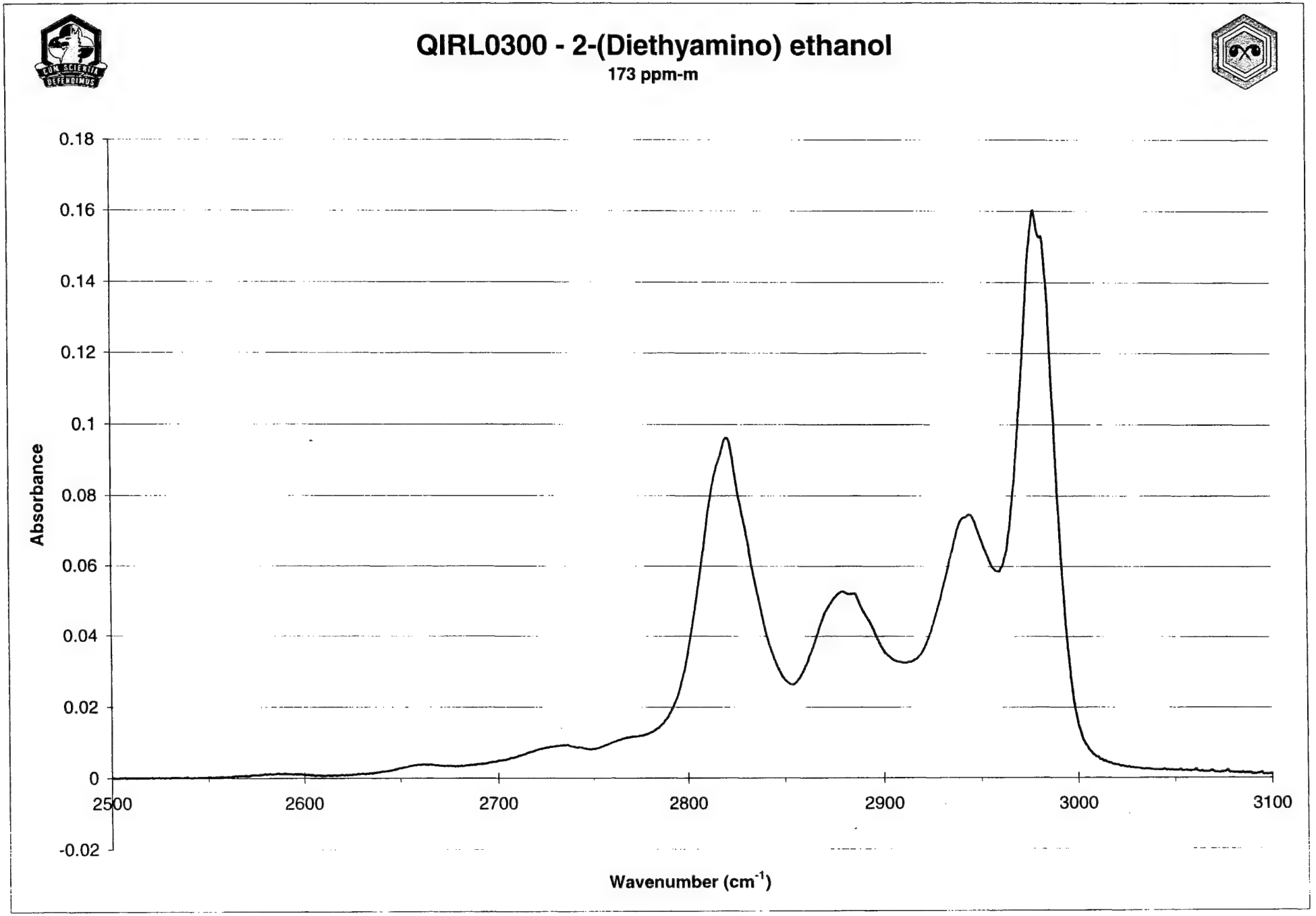

QIRL0290 - 2-(Diethyamino) ethanethiol
72 ppm-m
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

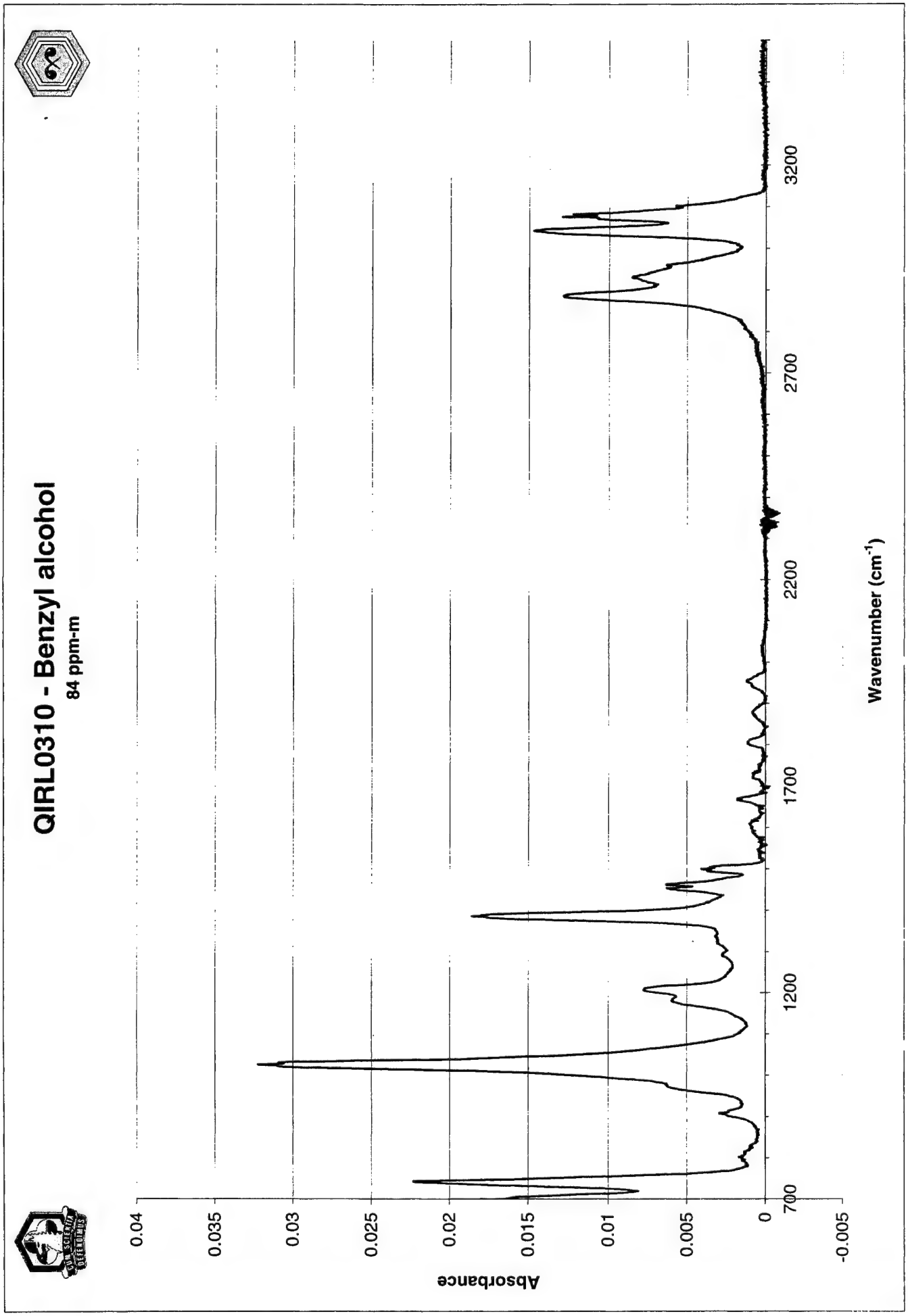

QIRL0290 - 2-(Diethyamino) ethanethiol
72 ppm-m
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

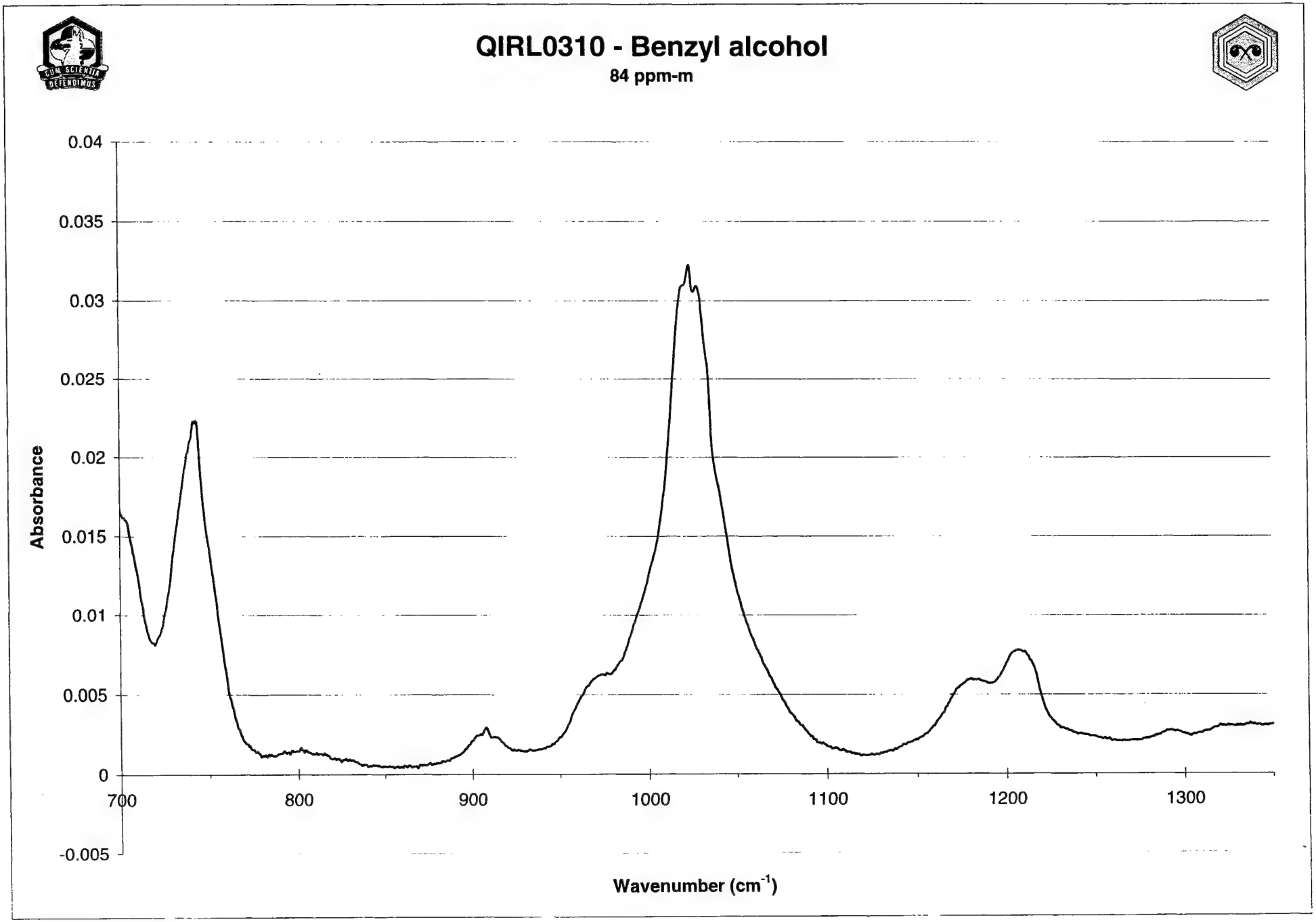

QIRL0290 - 2-(Diethyamino) ethanethiol
72 ppm-m
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

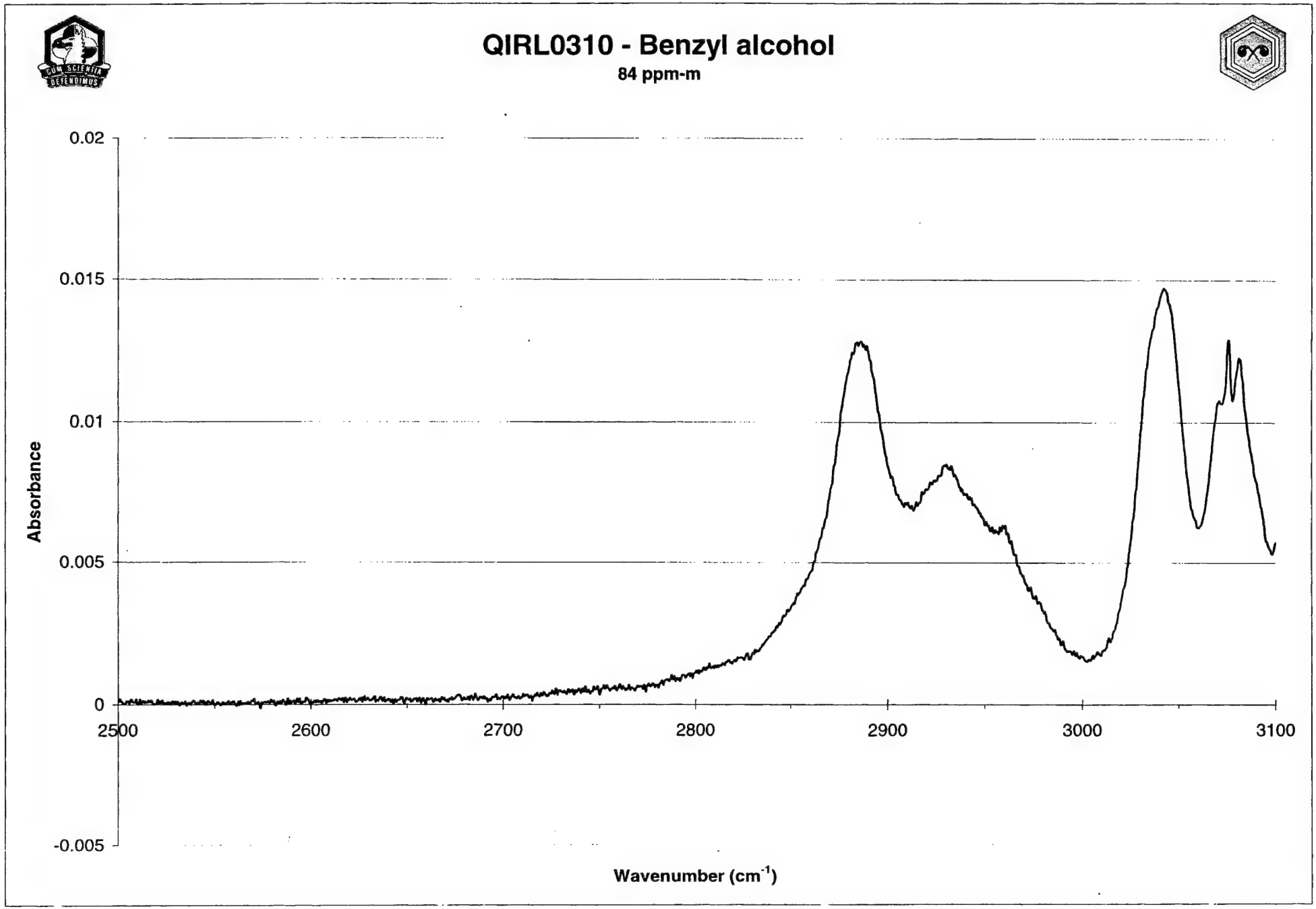

QIRL0300 - 2-(Diethyamino) ethanol
173 ppm-m
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
-0.02
Absorbance
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

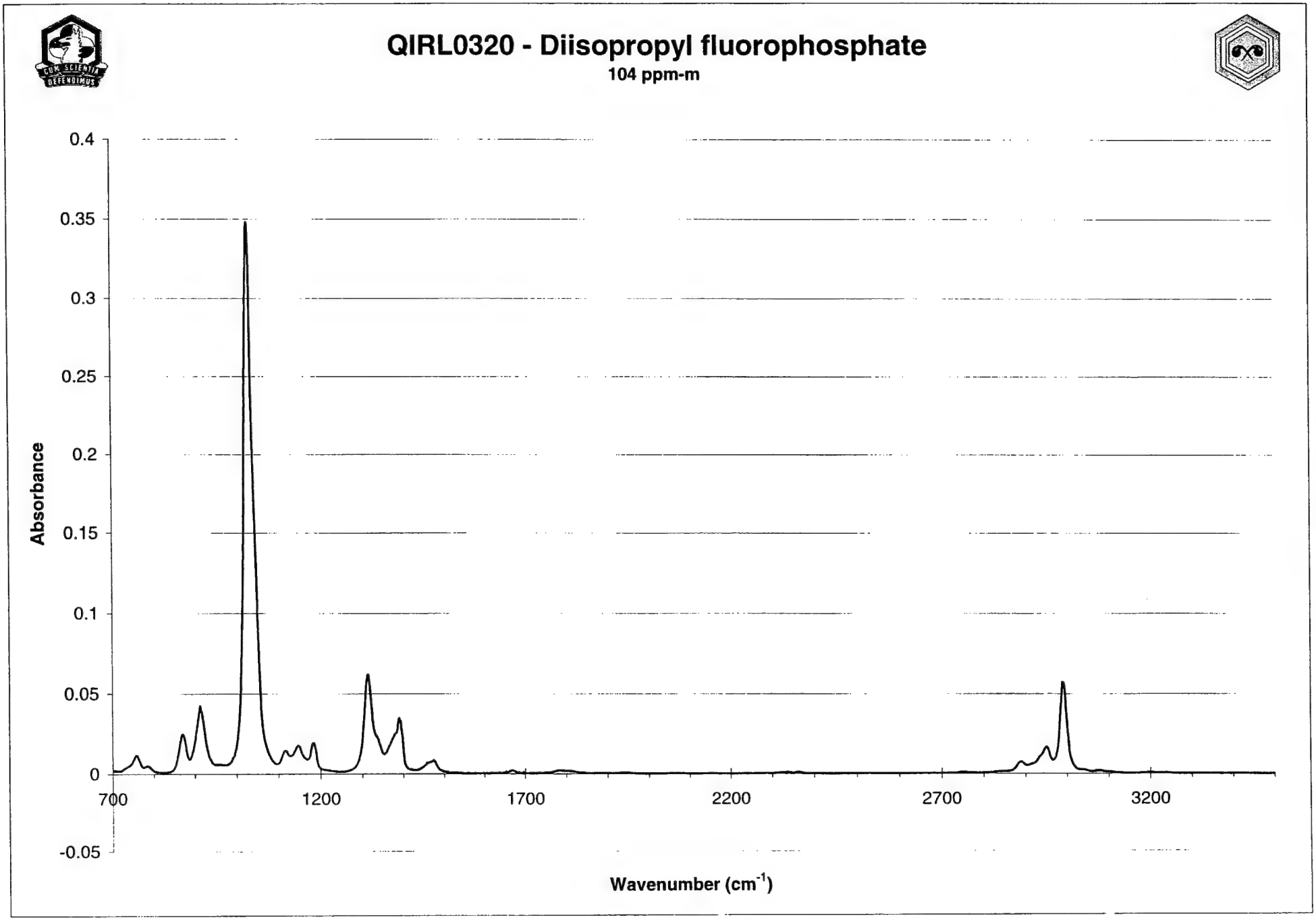

QIRL0300 - 2-(Diethyamino) ethanol
173 ppm-m
Absorbance
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

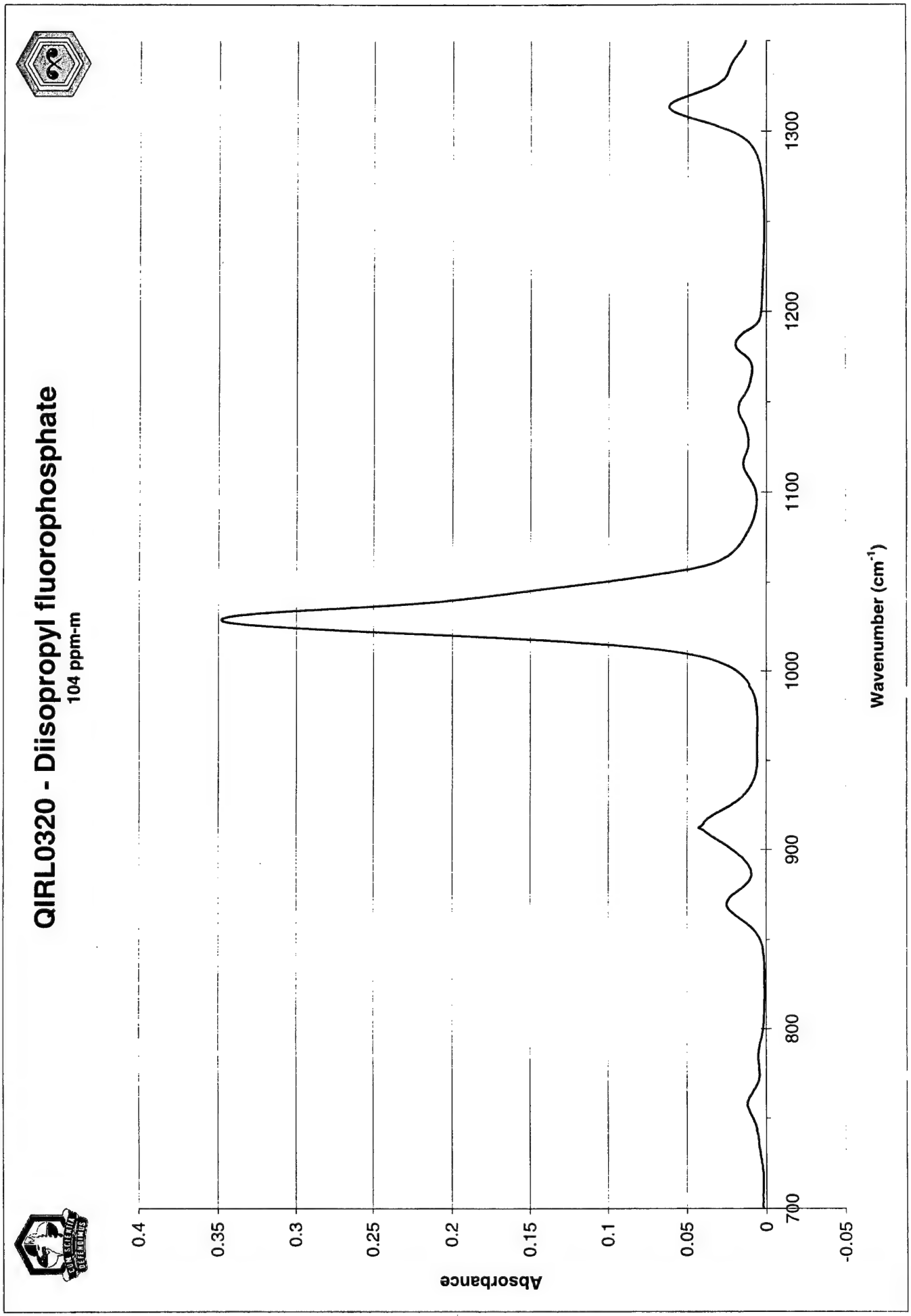

QIRL0300 - 2-(Diethyamino) ethanol
173 ppm-m
Absorbance
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
-0.02
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

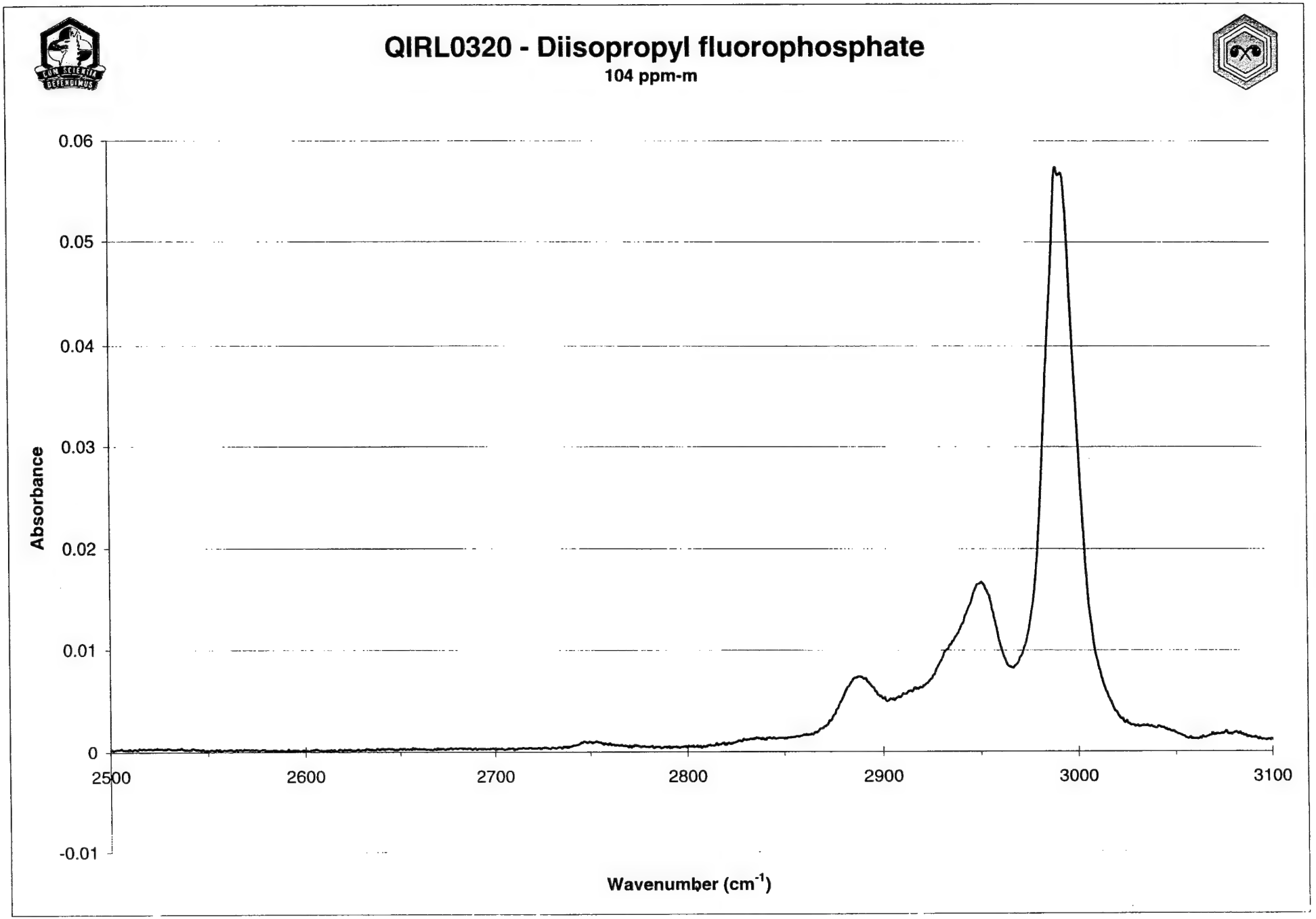

QIRL0310 - Benzyl alcohol
84 ppm-m
Absorbance
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
-0.005
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

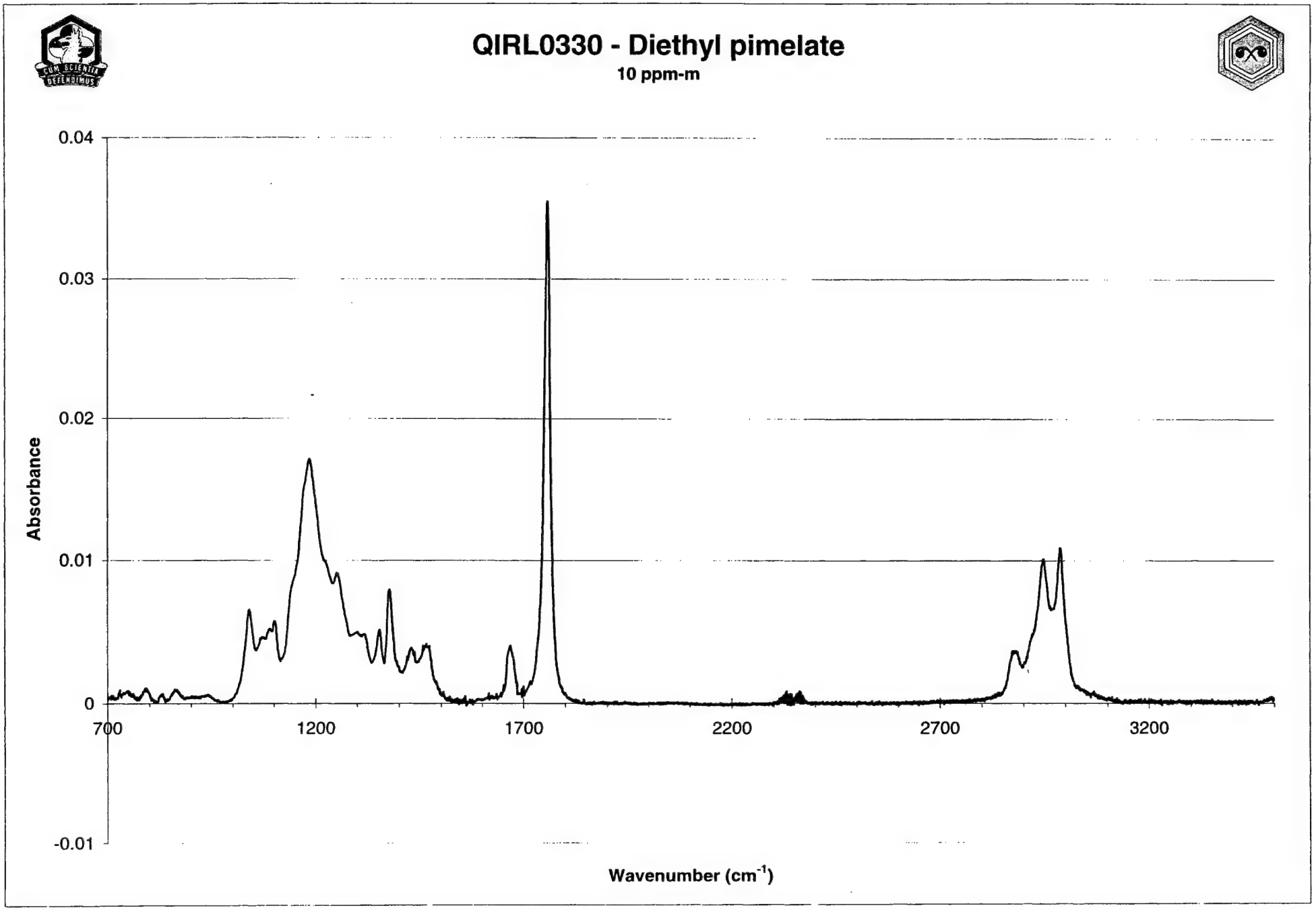

QIRL0310 - Benzyl alcohol
84 ppm-m
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

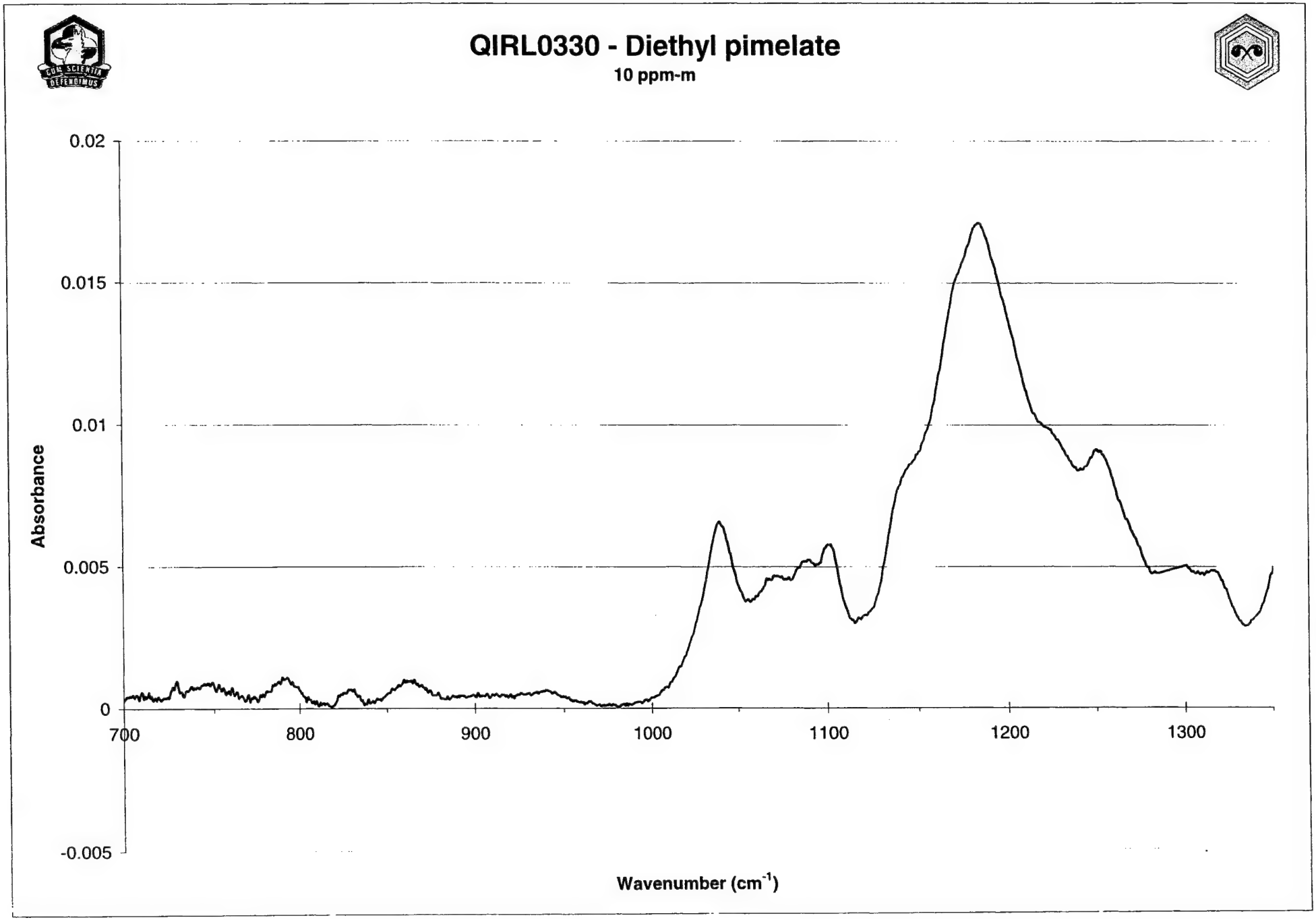

QIRL0310 - Benzyl alcohol
84 ppm-m
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

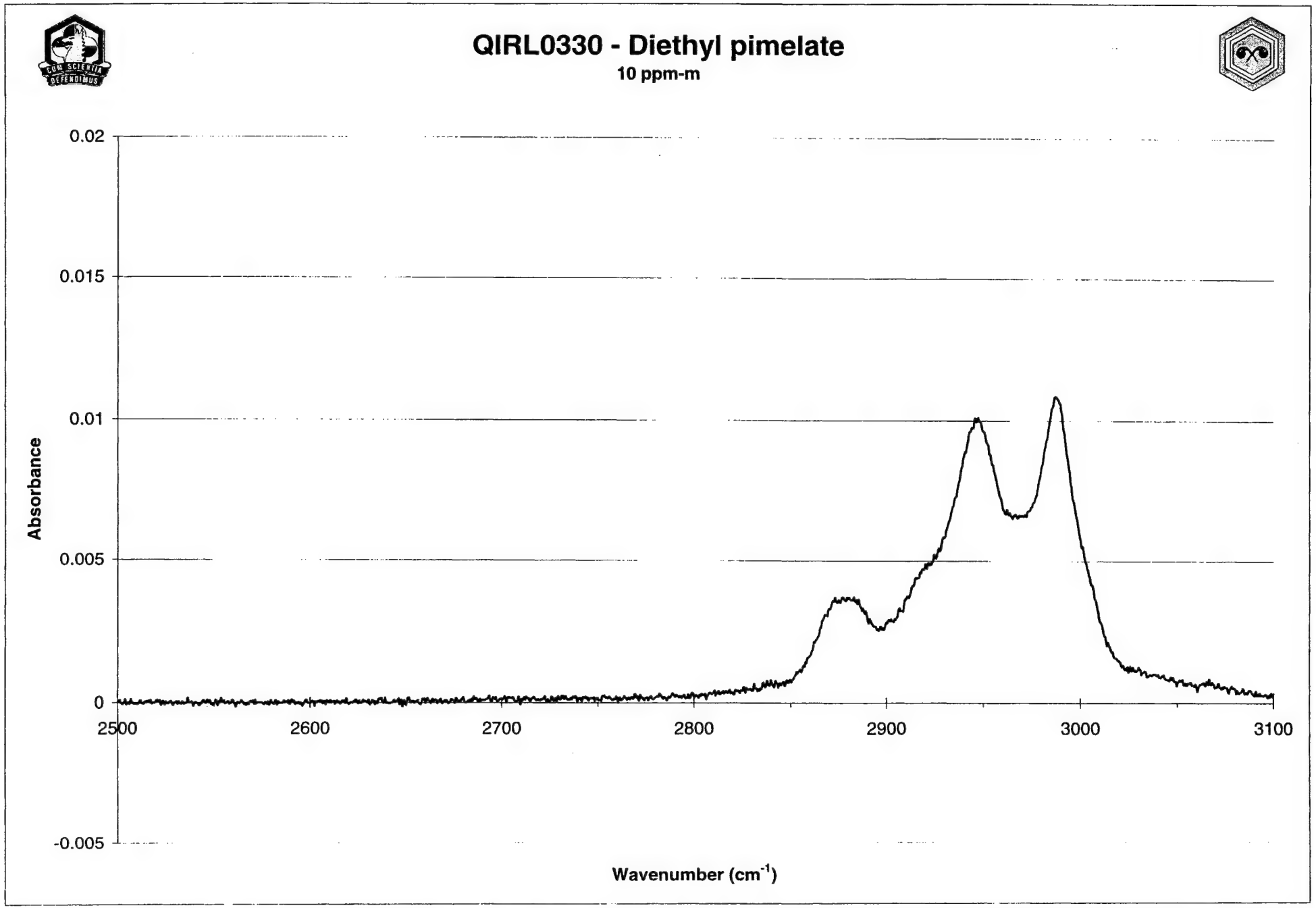

QIRL0320 - Diisopropyl fluorophosphate
104 ppm-m
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-0.05
Absorbance
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

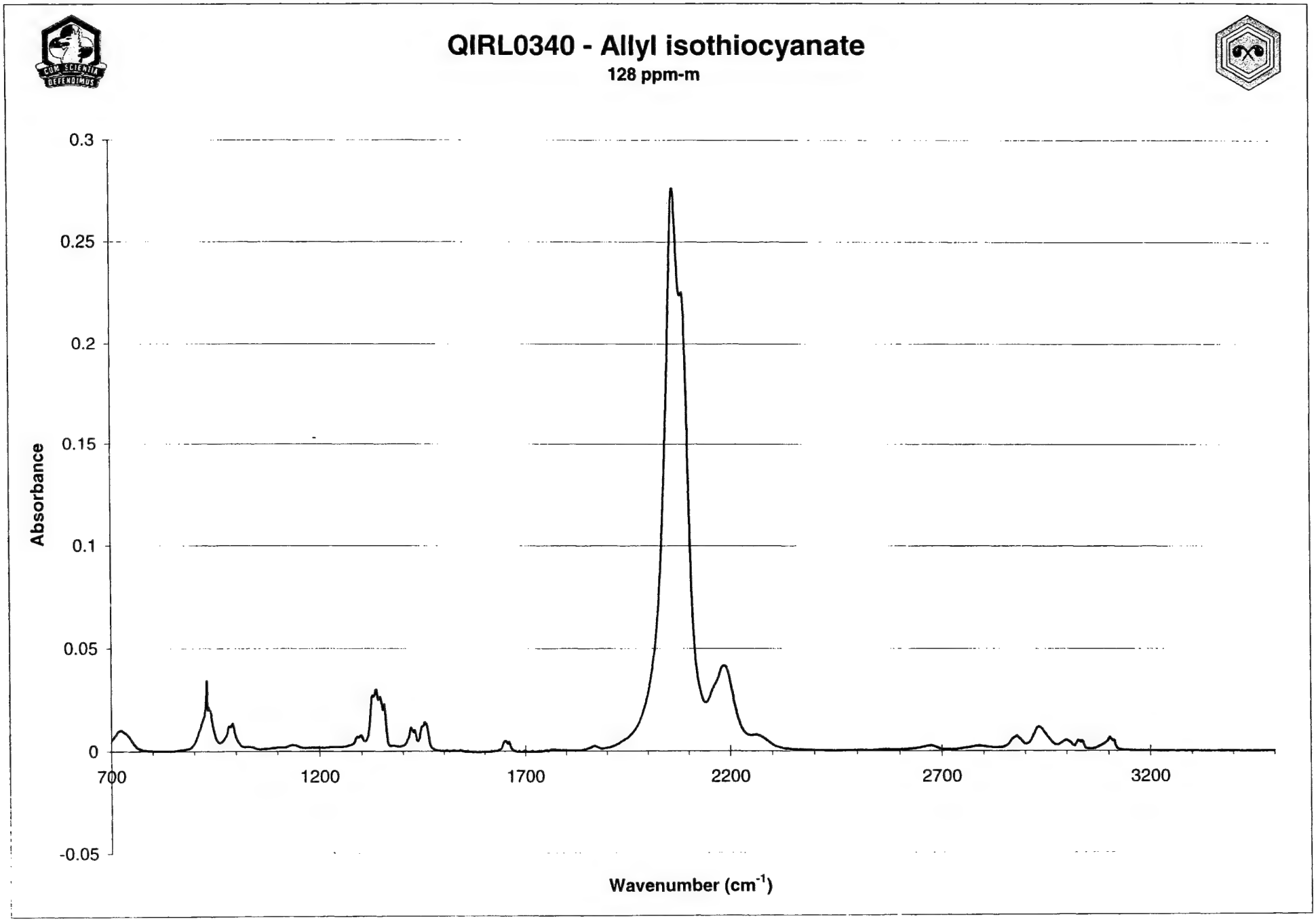

QIRL0320 - Diisopropyl fluorophosphate
104 ppm-m
Absorbance
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-0.05
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

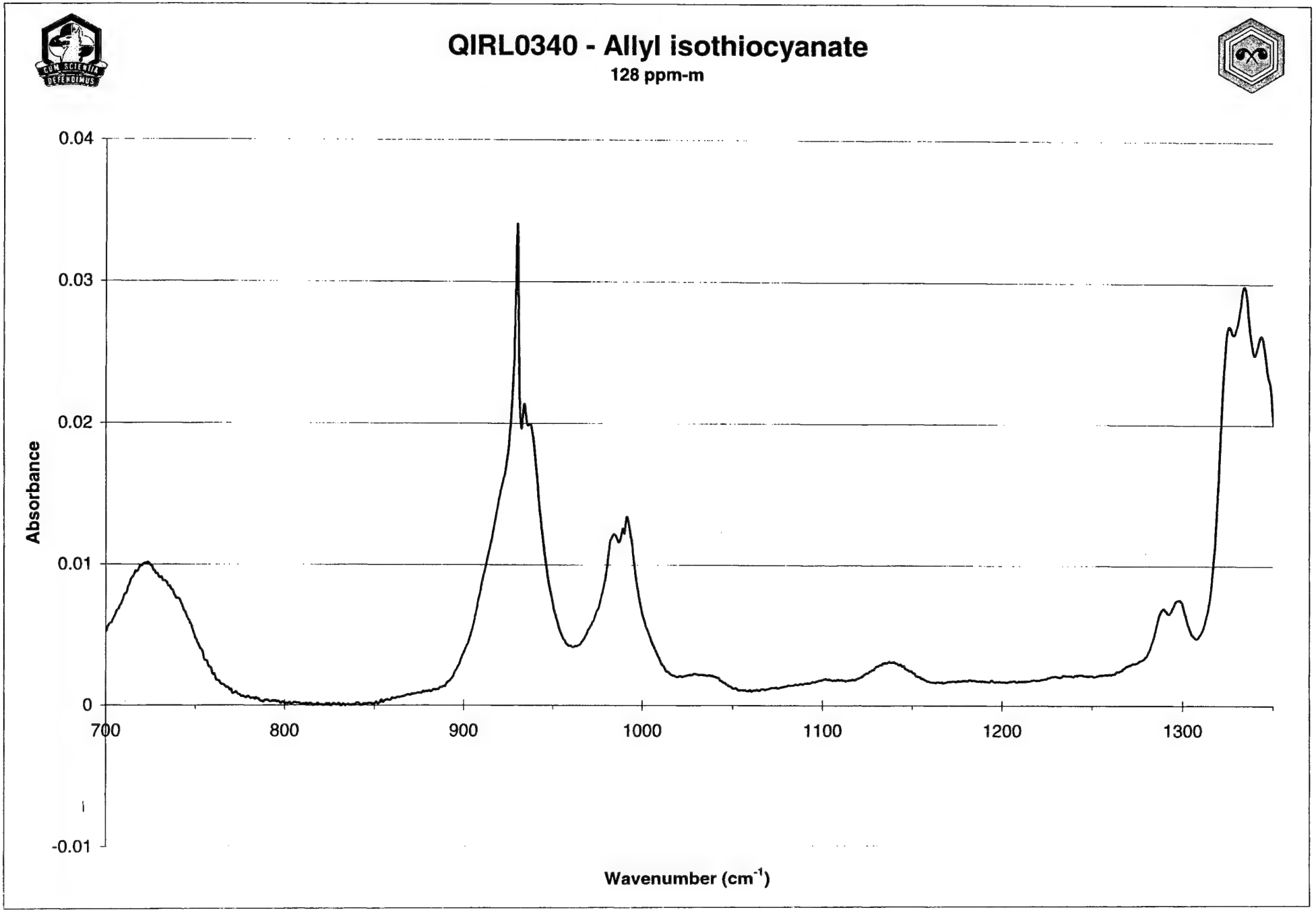

QIRL0320 - Diisopropyl fluorophosphate
104 ppm-m
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

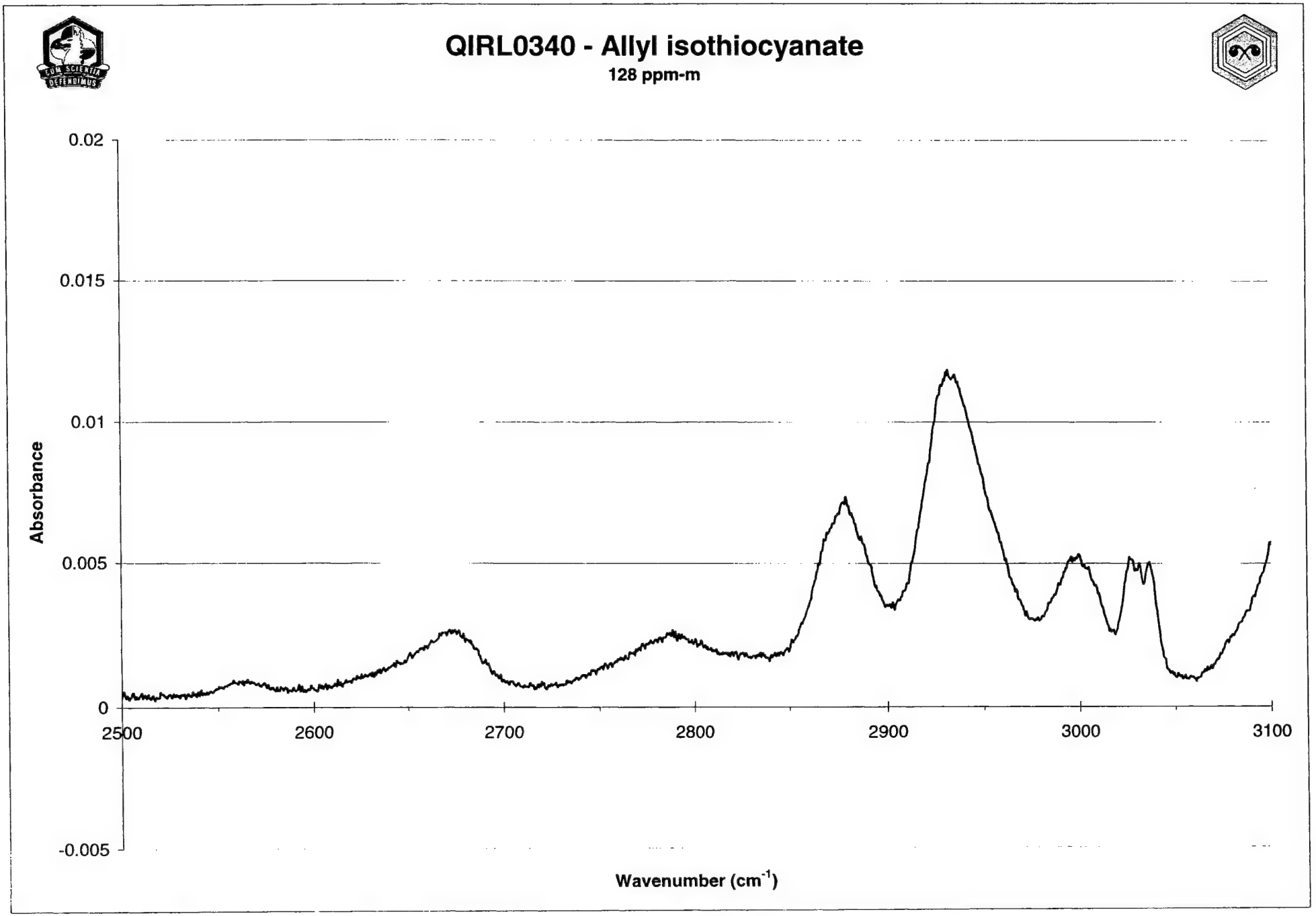

QIRL0330 - Diethyl pimelate
10 ppm-m
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

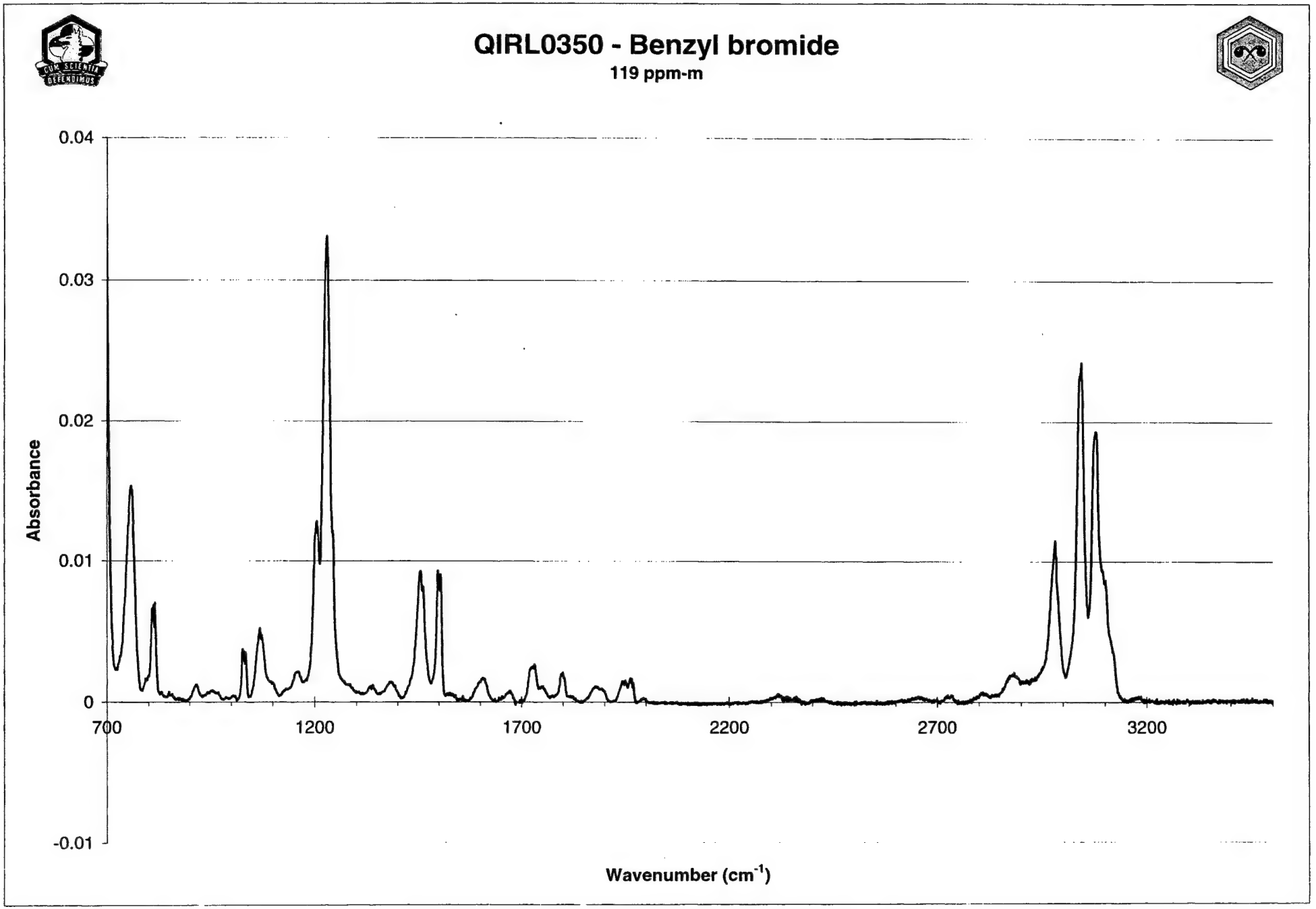

QIRL0330 - Diethyl pimelate
10 ppm-m
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

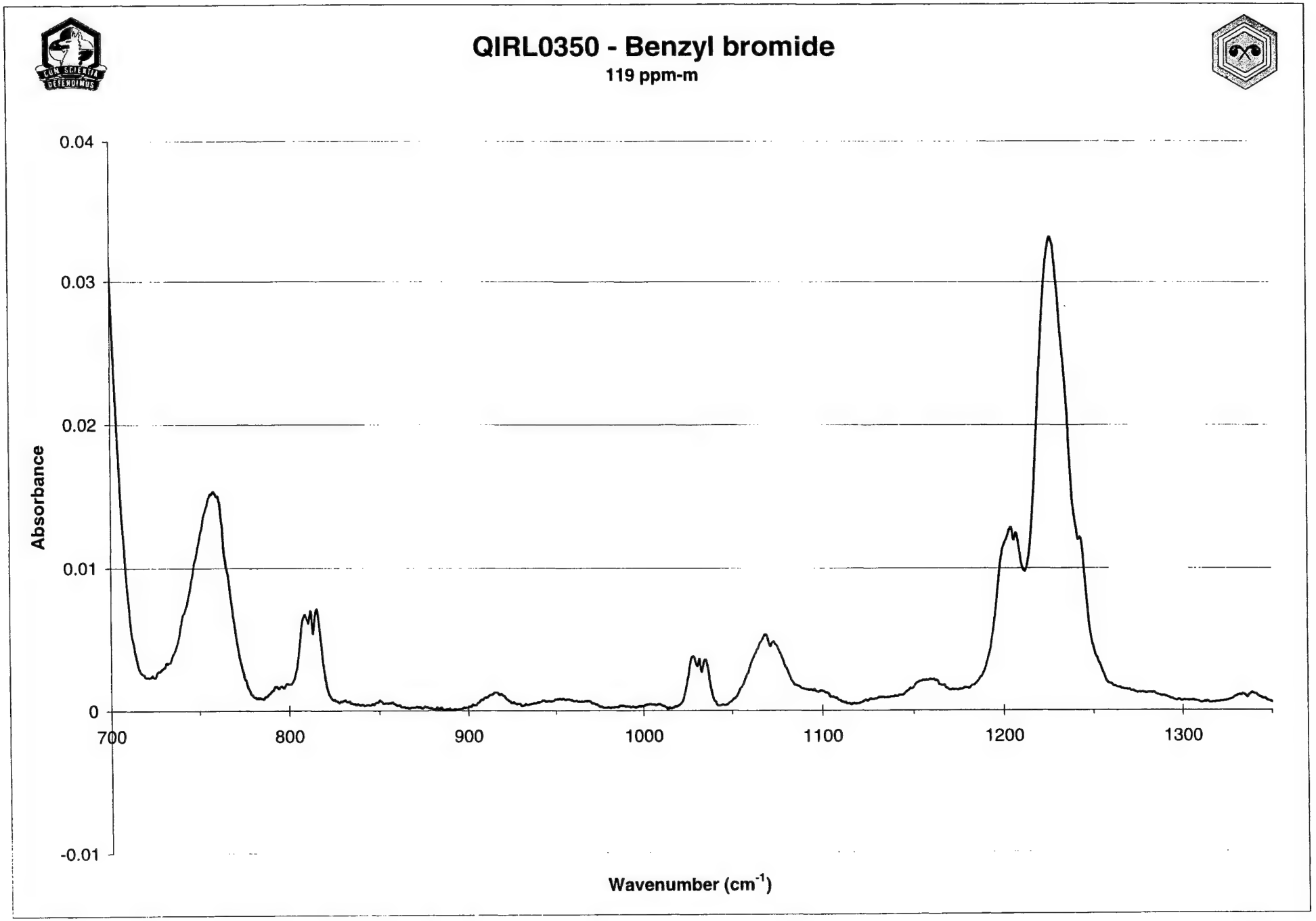

QIRL0330 - Diethyl pimelate
10 ppm-m
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

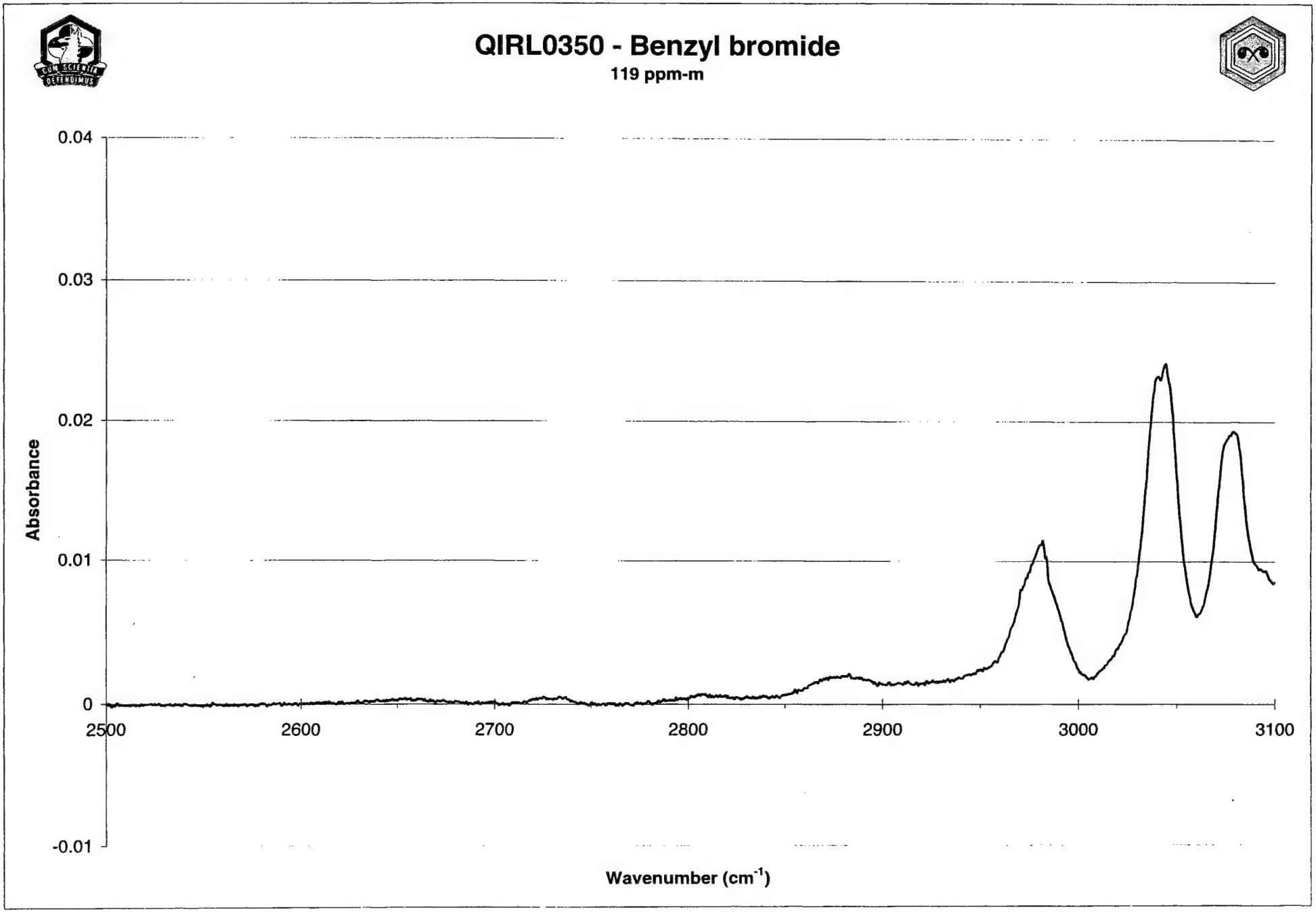

QIRL0340 - Allyl isothiocyanate
128 ppm-m
0.3
0.25
0.2
0.15
0.1
0.05
0
-0.05
Absorbance
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

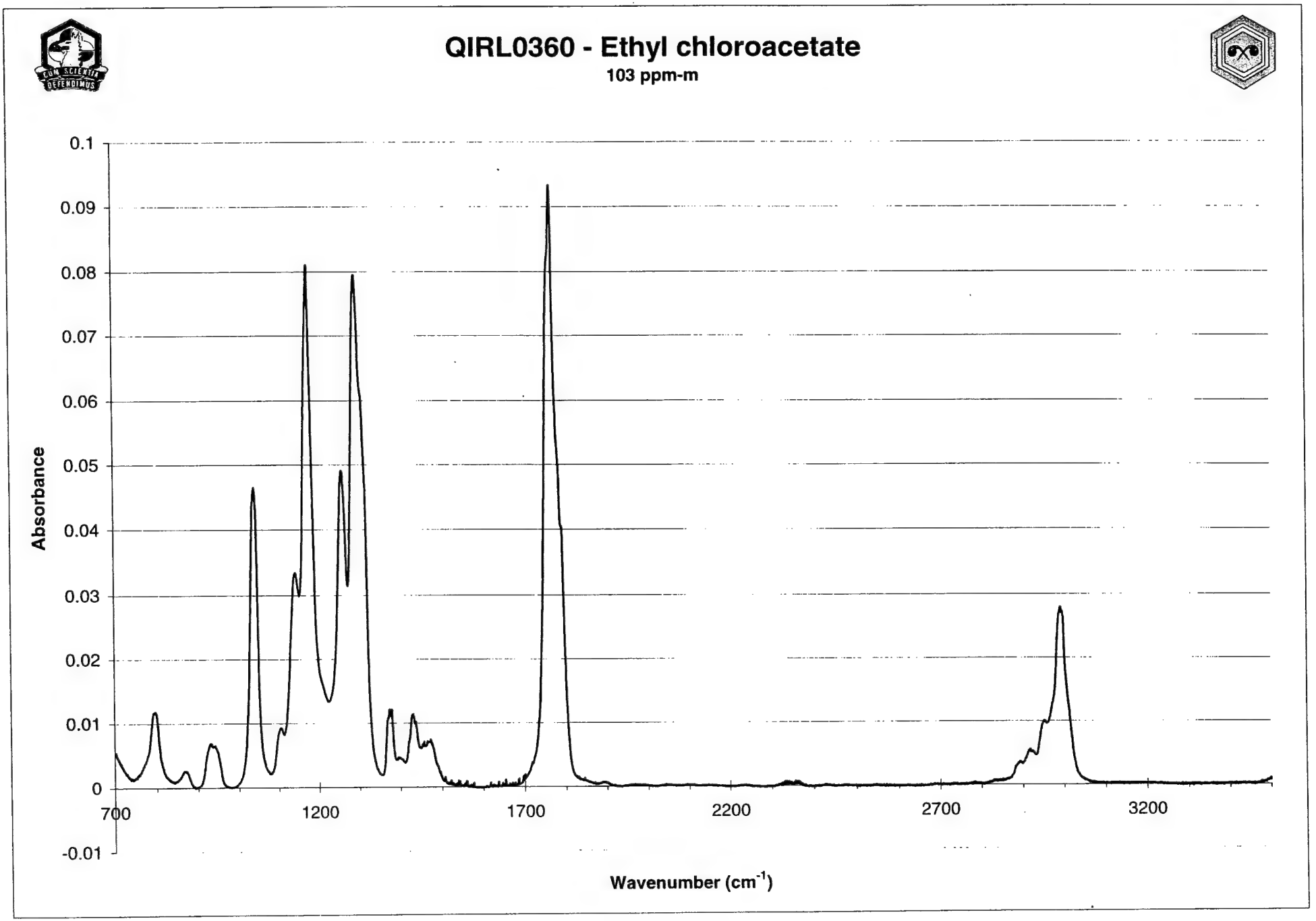

QIRL0340 - Allyl isothiocyanate
128 ppm-m
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

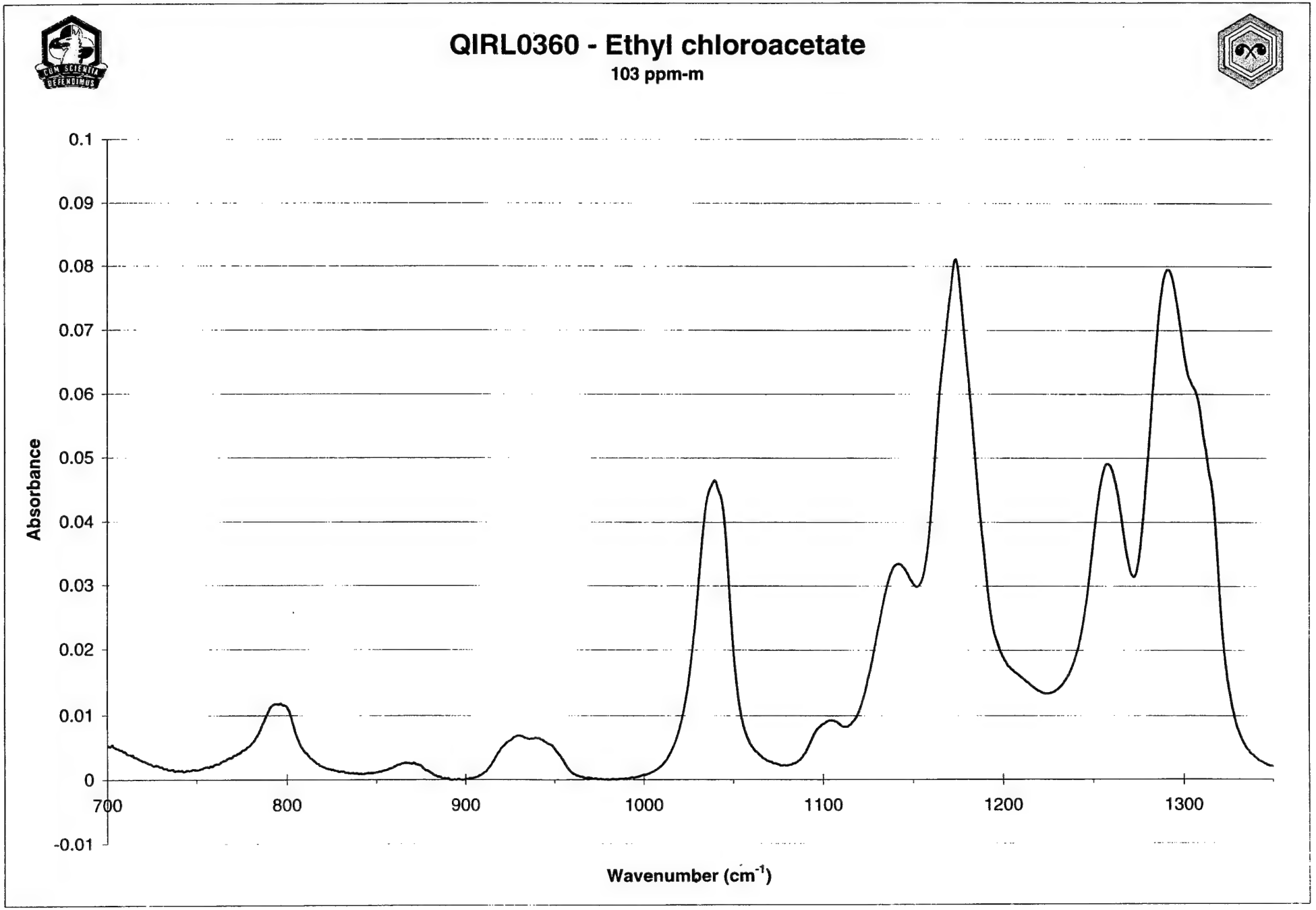

QIRL0340 - Allyl isothiocyanate
128 ppm-m
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

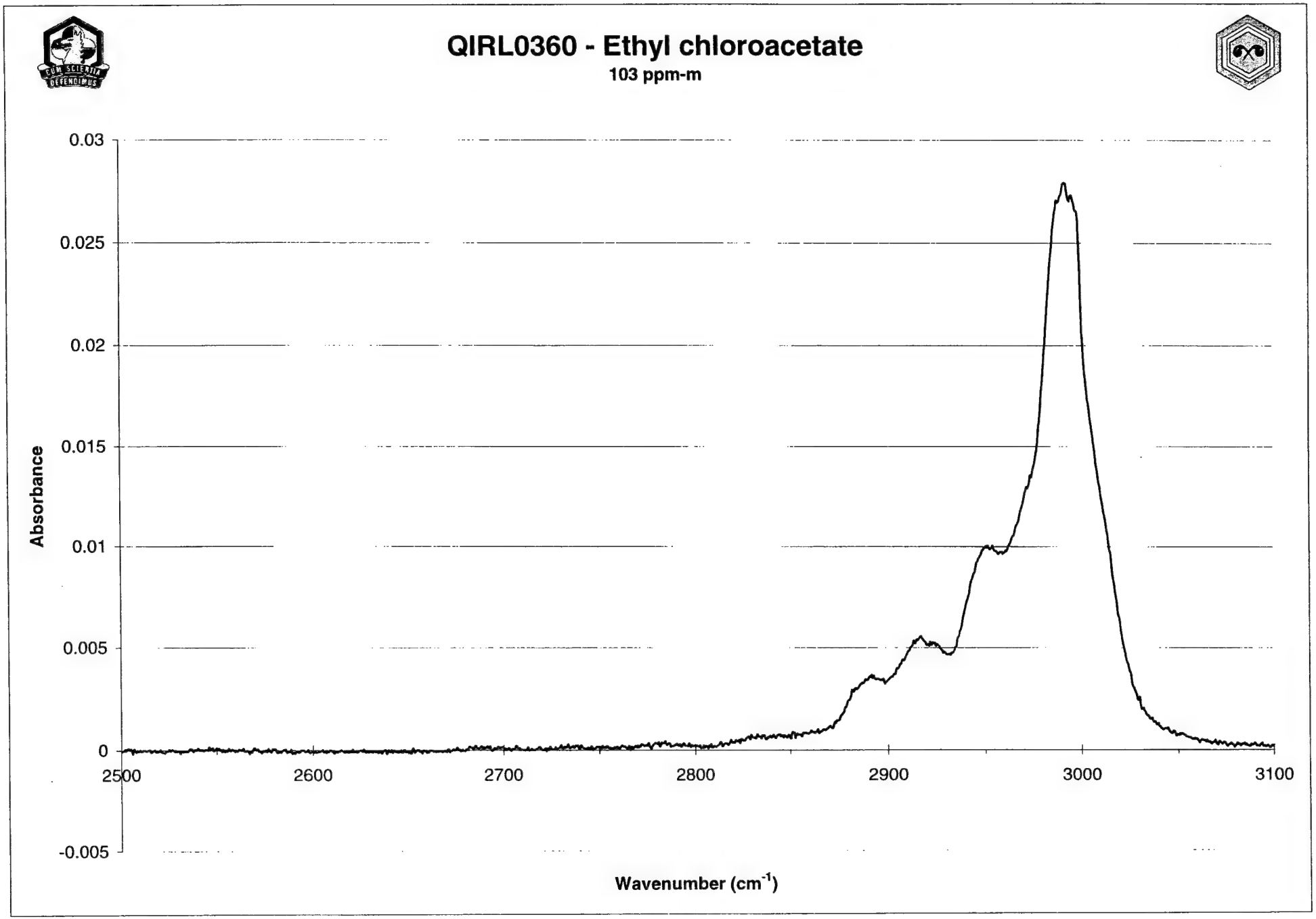

QIRL0350 - Benzyl bromide
119 ppm-m
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

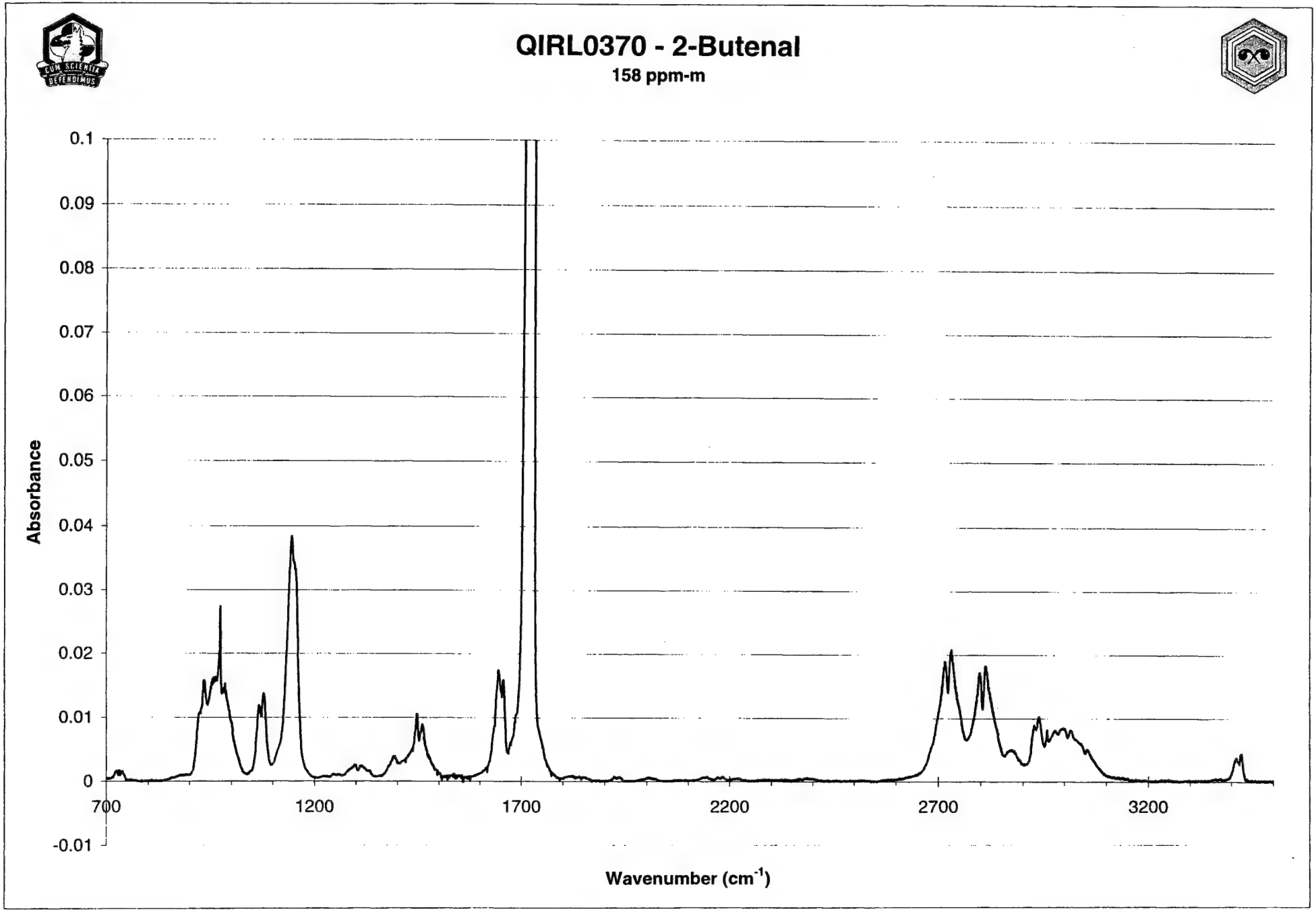

QIRL0350 - Benzyl bromide
119 ppm-m
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

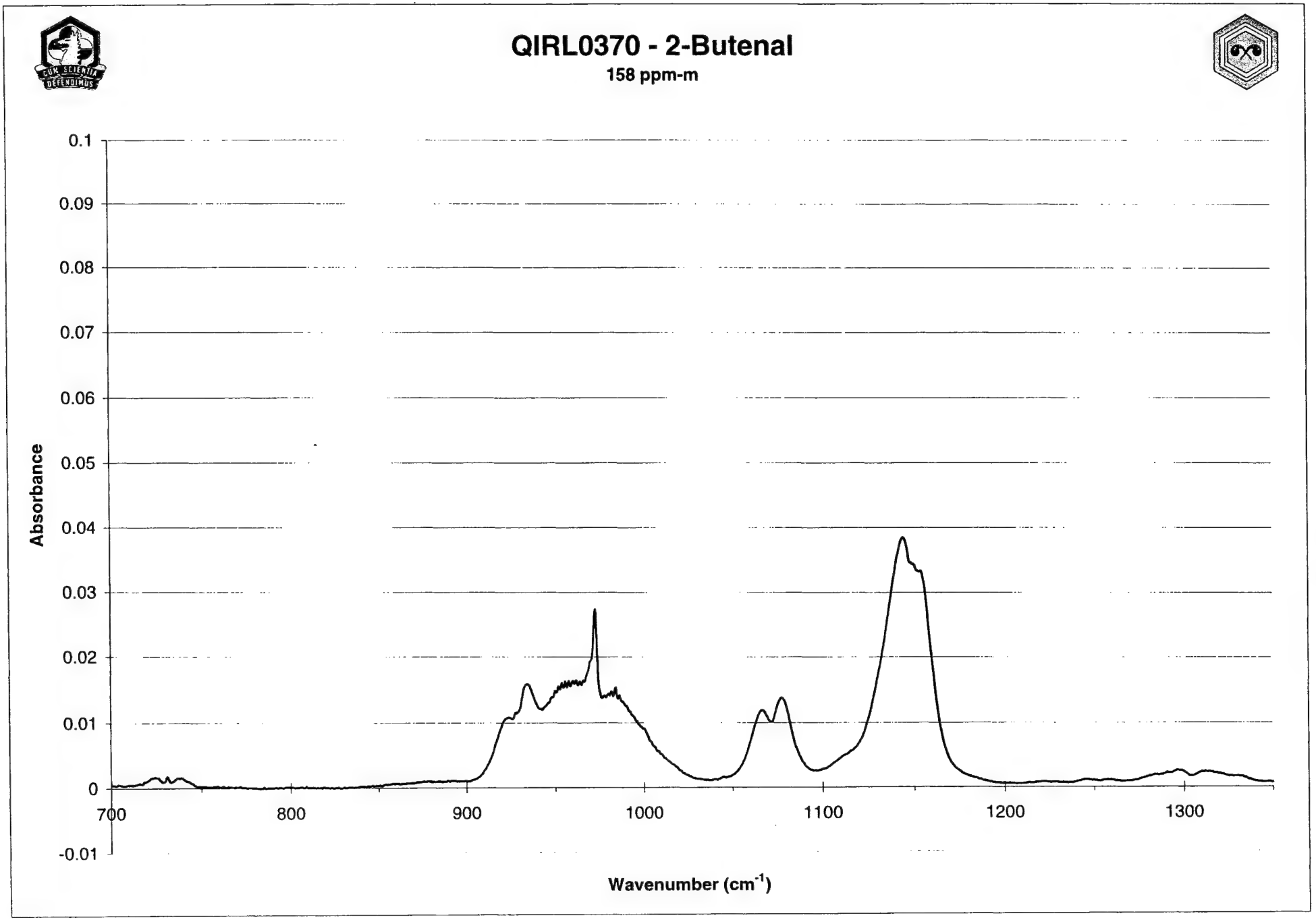

QIRL0350 - Benzyl bromide
119 ppm-m
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

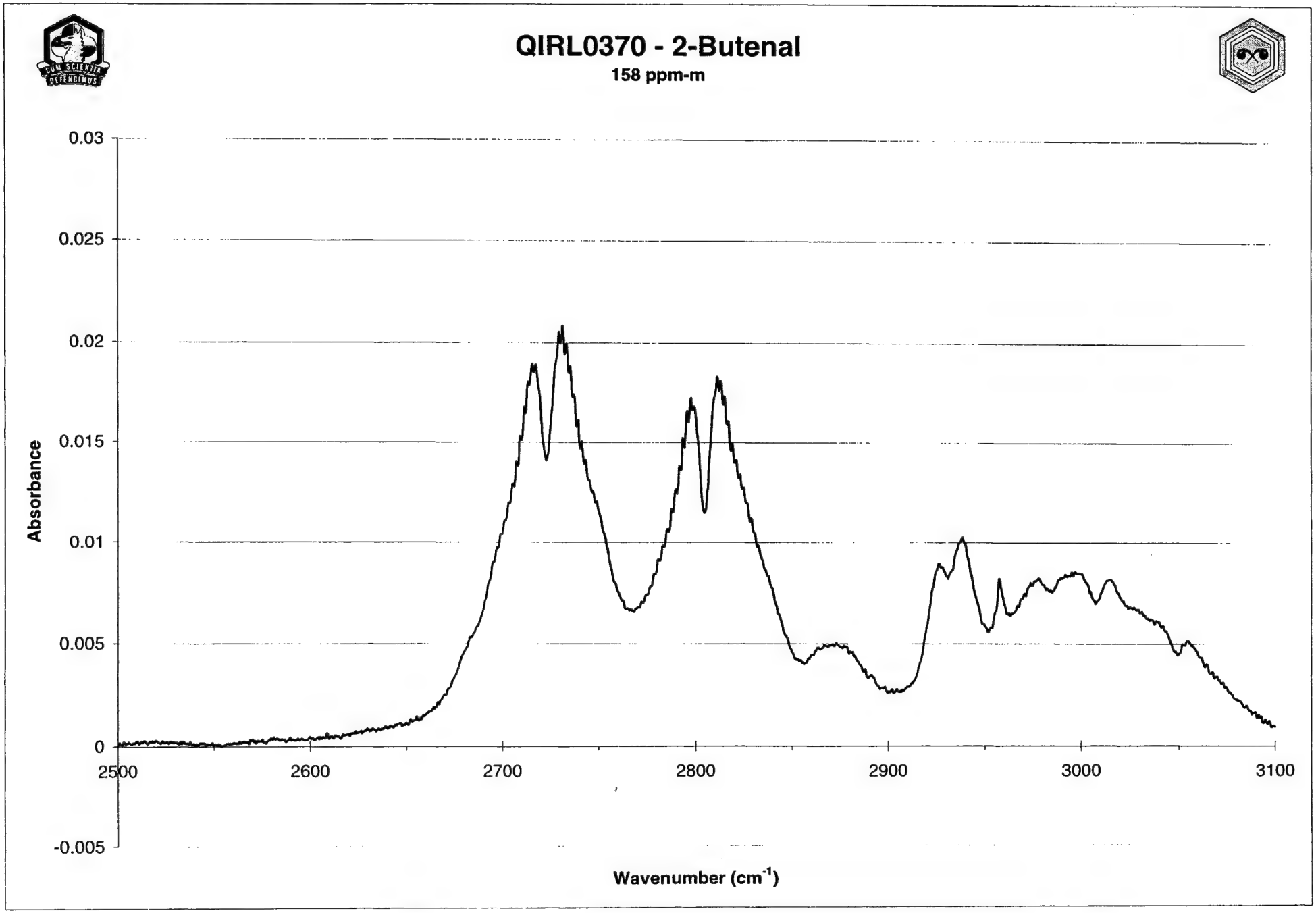

QIRL0360 - Ethyl chloroacetate
103 ppm-m
Absorbance
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

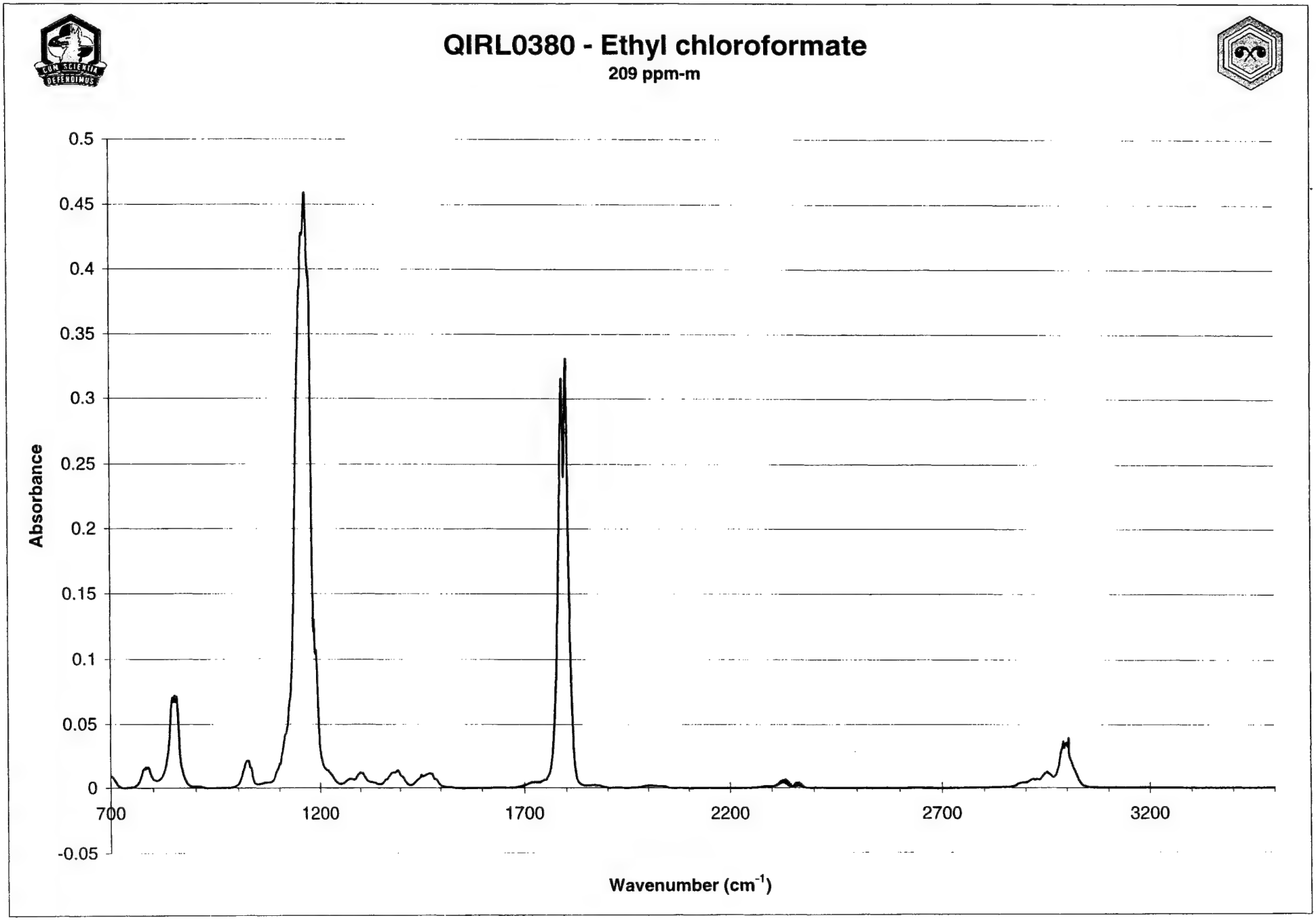

QIRL0360 - Ethyl chloroacetate
103 ppm-m
Absorbance
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

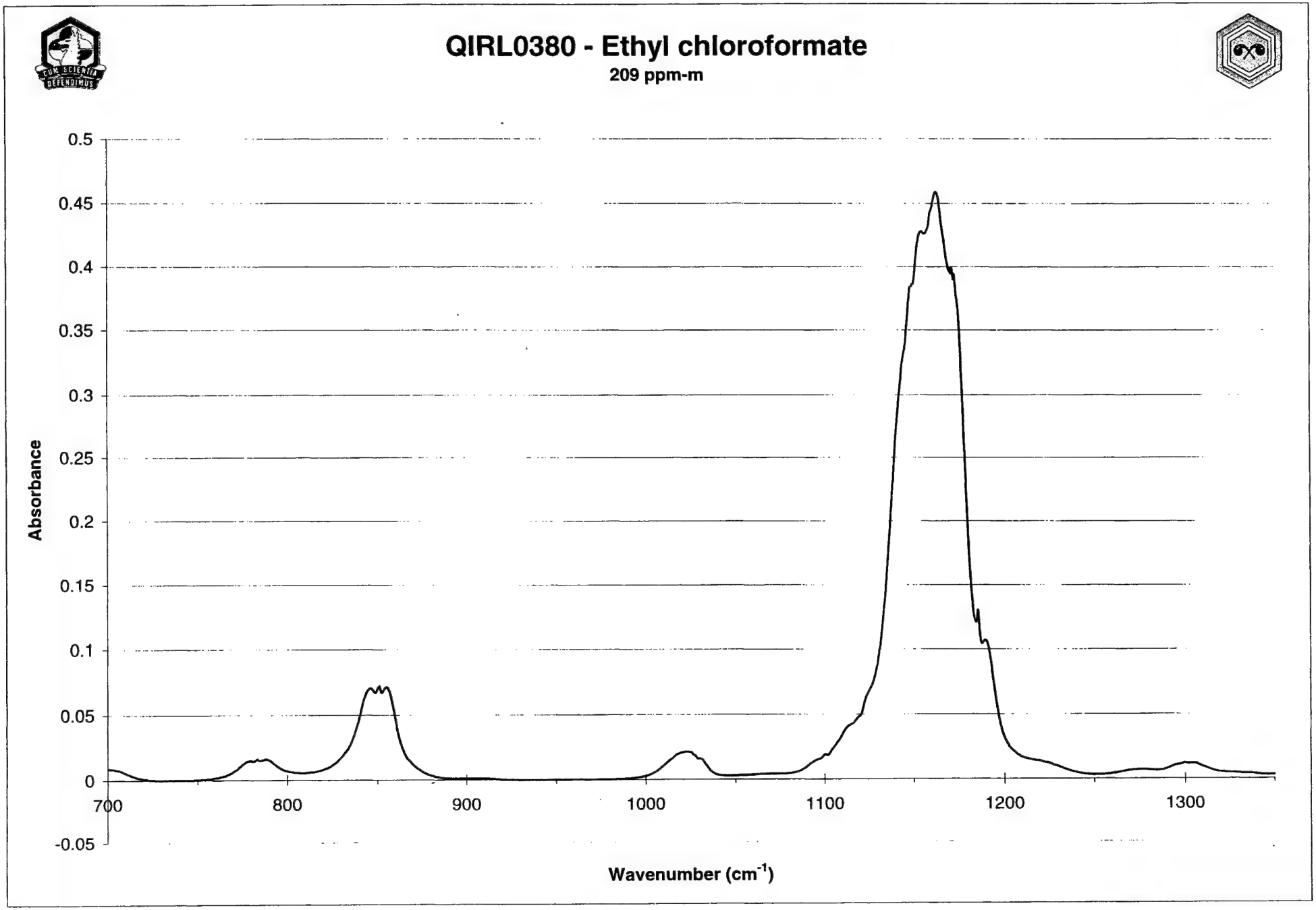

QIRL0360 - Ethyl chloroacetate
103 ppm-m
0.03
0.025
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

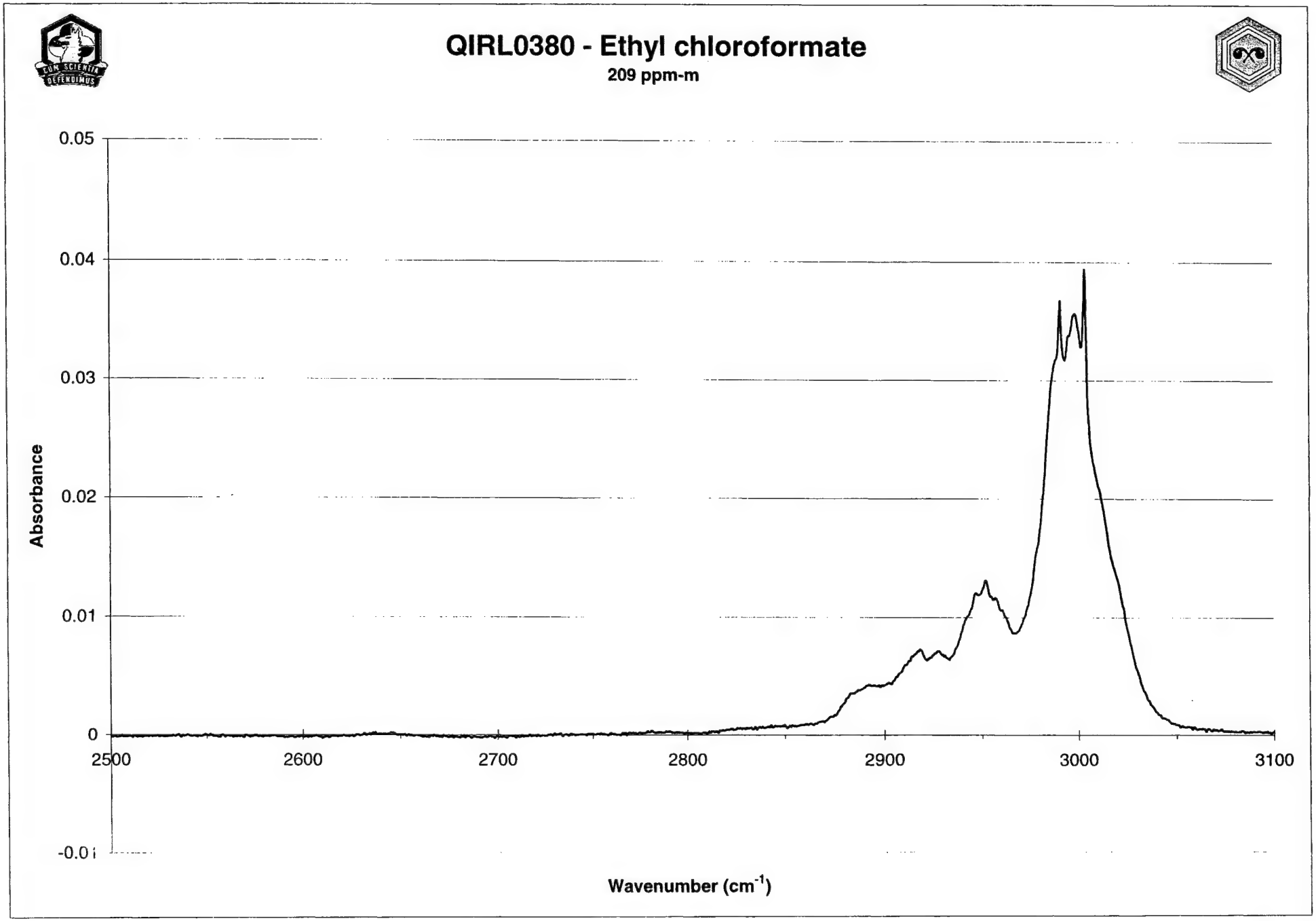

QIRL0370 - 2-Butenal
158 ppm-m
Absorbance
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

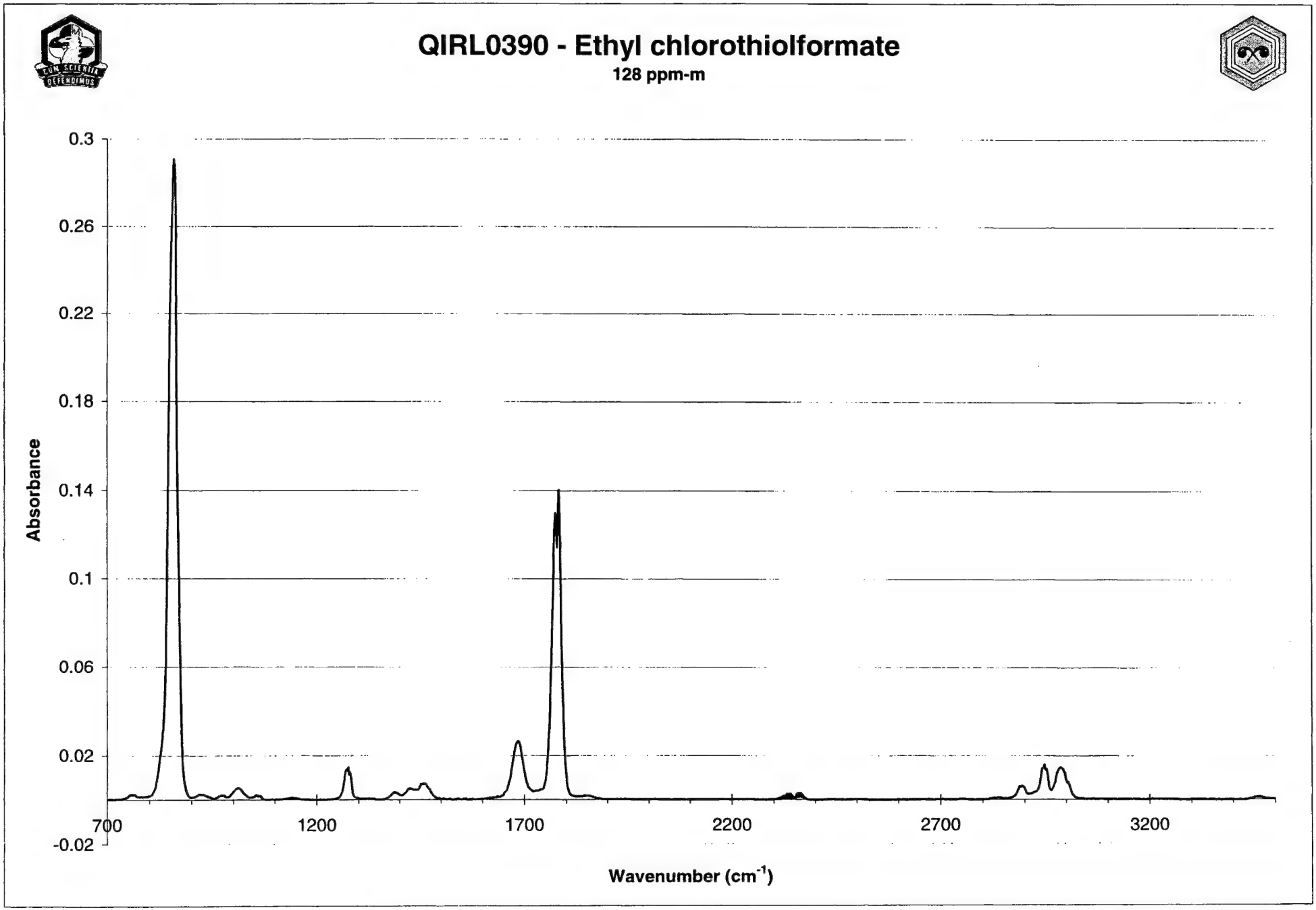

QIRL0370 - 2-Butenal
158 ppm-m
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

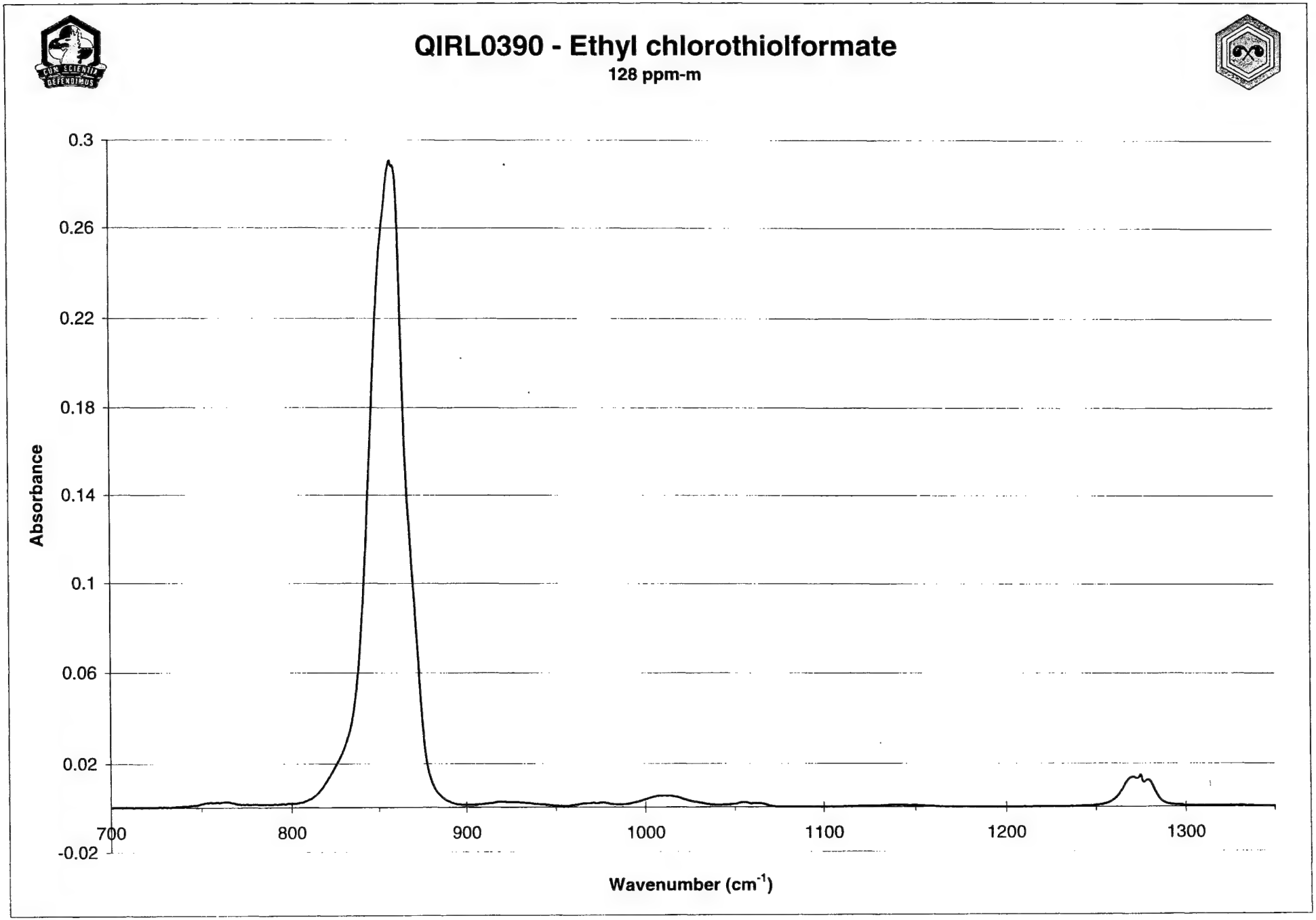

QIRL0370 - 2-Butenal
158 ppm-m
0.03
0.025
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

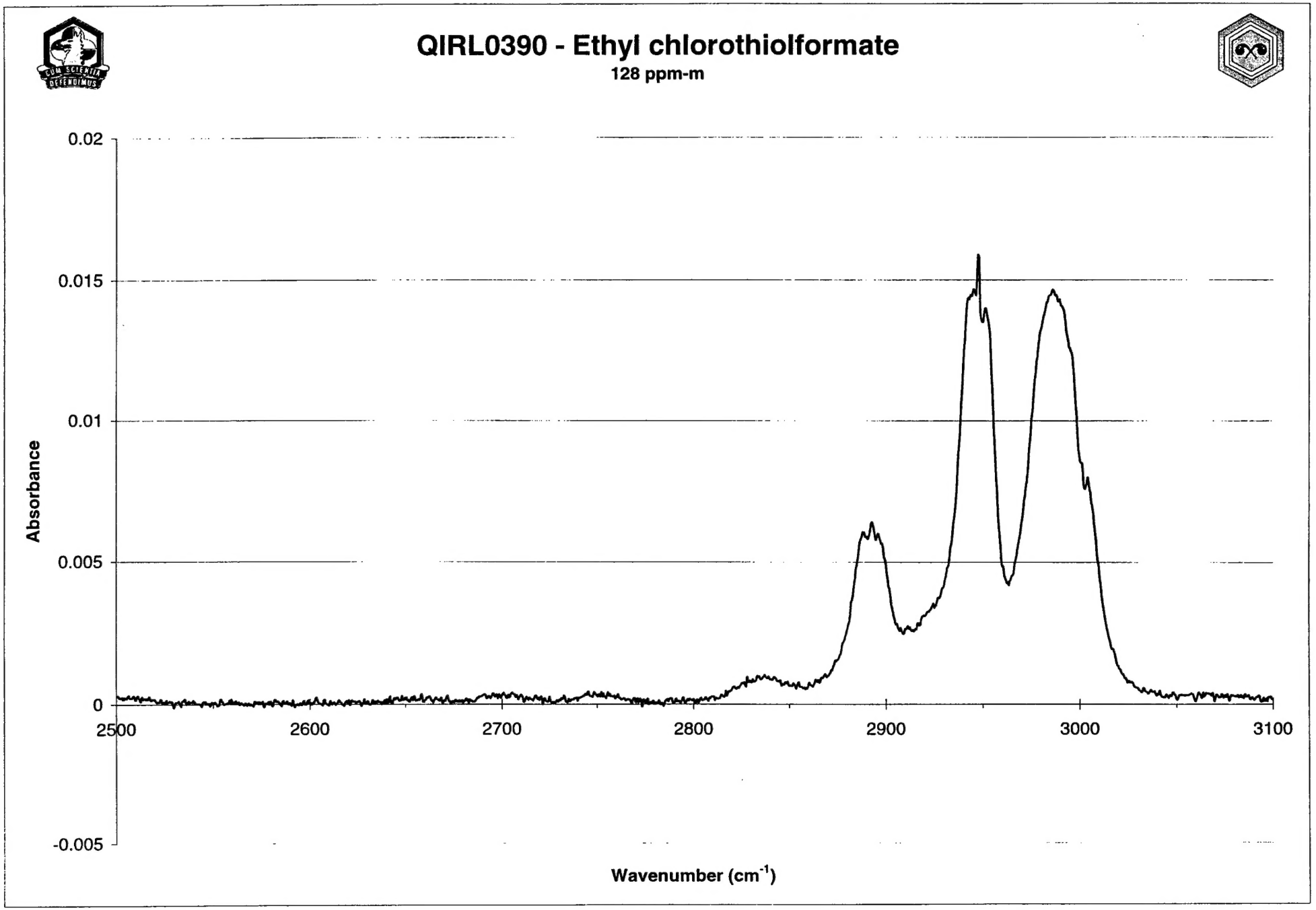

QIRL0380 - Ethyl chloroformate
209 ppm-m
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-0.05
Absorbance
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)

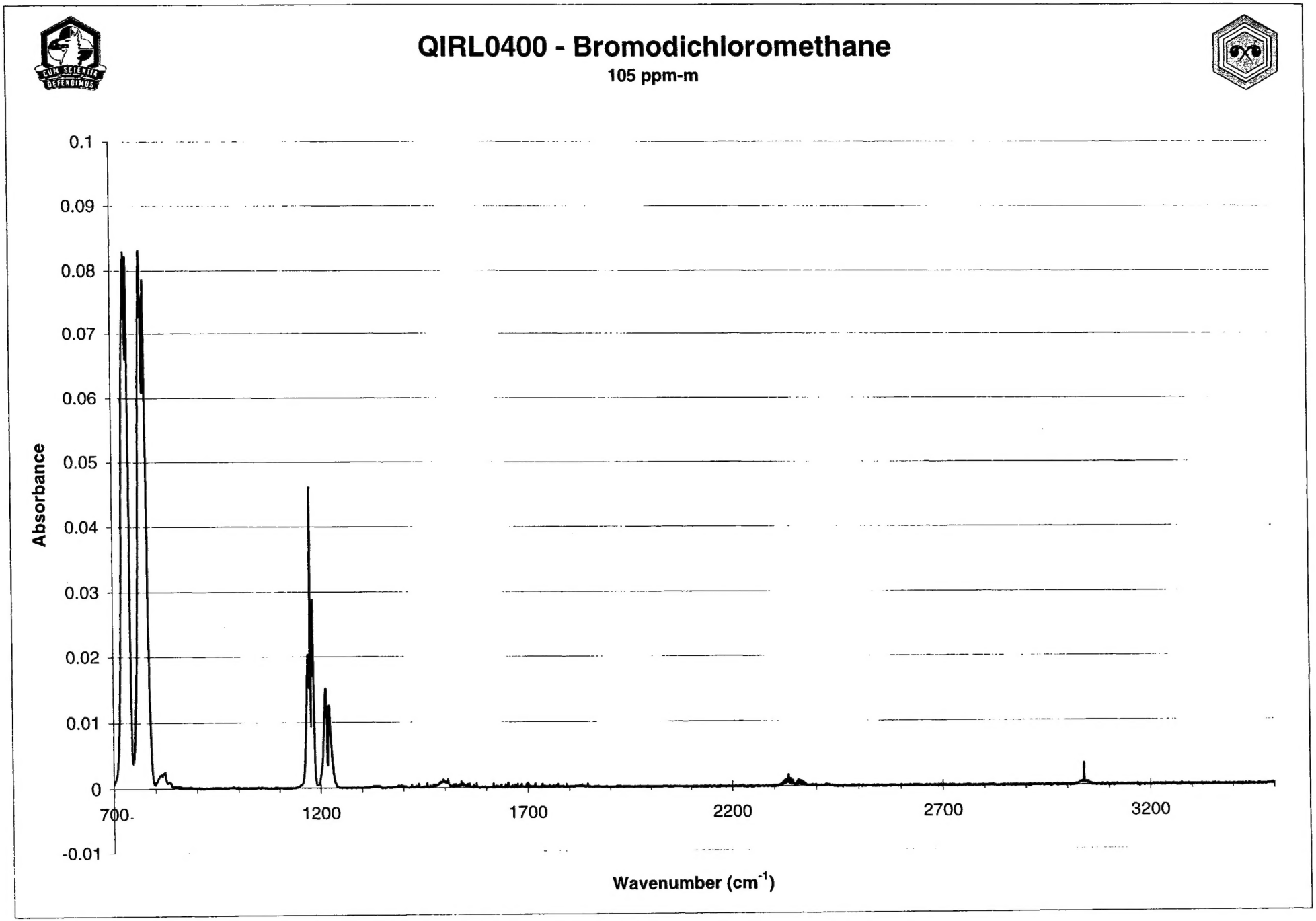

QIRL0380 - Ethyl chloroformate
209 ppm-m
Absorbance
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-0.05
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)

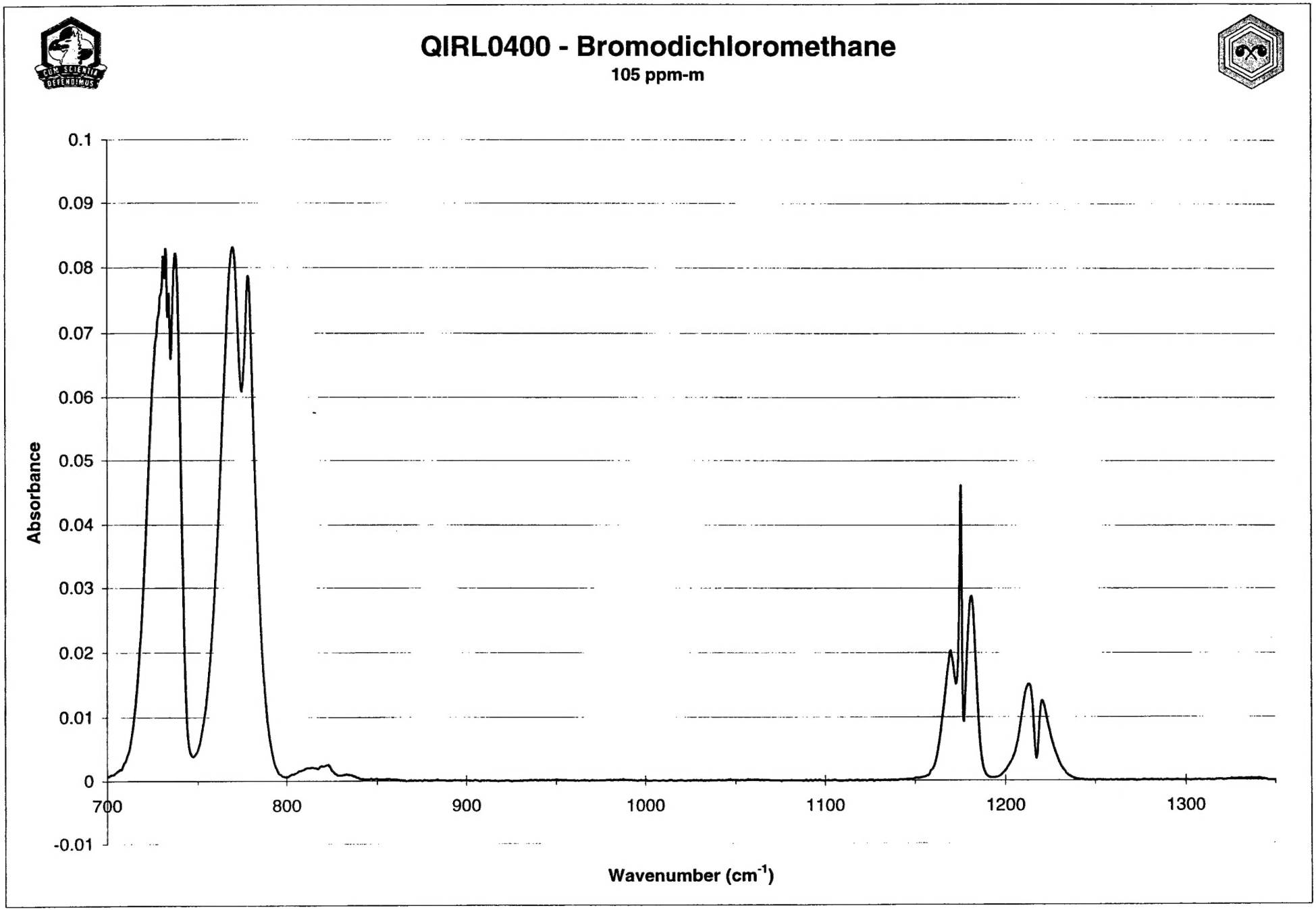

QIRL0380 - Ethyl chloroformate
209 ppm-m
0.05
0.04
0.03
0.02
0.01
0
-0.01
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)

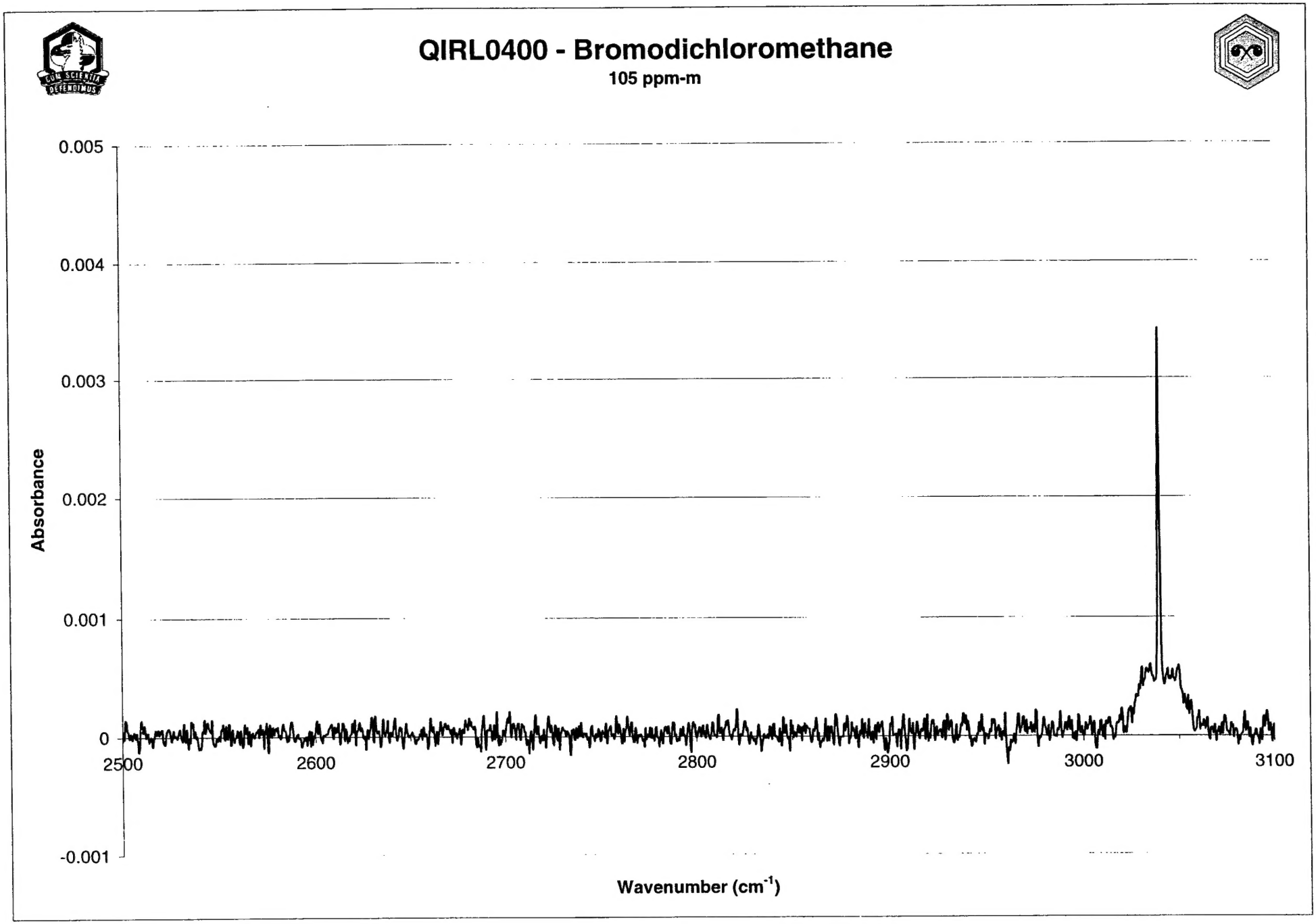

QIRL0390 - Ethyl chlorothiolformate
128 ppm-m
0.3
0.26
0.22
0.18
0.14
0.1
0.06
0.02
-0.02
Absorbance
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)


QIRL0390 - Ethyl chlorothiolformate
128 ppm-m
0.3
0.26
0.22
0.18
0.14
0.1
0.06
0.02
-0.02
Absorbance
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)


QIRL0390 - Ethyl chlorothiolformate
128 ppm-m
0.02
0.015
0.01
0.005
0
-0.005
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)


QIRL0400 - Bromodichloromethane
105 ppm-m
Absorbance
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
700
1200
1700
2200
2700
3200
Wavenumber (cm-1)


QIRL0400 - Bromodichloromethane
105 ppm-m
Absorbance
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
700
800
900
1000
1100
1200
1300
Wavenumber (cm-1)


QIRL0400 - Bromodichloromethane
105 ppm-m
0.005
0.004
0.003
0.002
0.001
0
-0.001
Absorbance
2500
2600
2700
2800
2900
3000
3100
Wavenumber (cm-1)